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Changes in rheology and microstructure of bread dough

Thesis submitted in fulfilment of the requirements
for the degree of Doctor (PhD) in Applied Biological Sciences:
Chemistry

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Samenvatting

Tarwebloem is de enige bloemsoort waarmee een deeg kan worden gevormd dat beschikt over de gewenste visco-elastische eigenschappen in broodbereiding. Een tarwebloem deeg vertoont een unieke balans tussen viskeuze eigenschappen, welke toelaten dat het deeg rijst tijdens de fermentatie, en elastische eigenschappen, die het deeg verstevigen. In dit onderzoek was het de bedoeling om meer inzicht te verkrijgen over reologie en microstructuur van brooddeeg en de invloed hierop van de kwaliteit van de tarwebloem en de condities tijdens de deegverwerking.

In de literatuurstudie wordt een algemene introductie gegeven met betrekking tot de samenstelling van tarwebloem, deegvorming en de microstructuur van deeg. Verder wordt een overzicht gegeven van de experimentele technieken aangewend voor het bepalen van de reologische eigenschappen van deeg gevolgd door de theoretische achtergrond van de reologische eigenschappen die werden toegepast in dit onderzoek, meer bepaald dynamische oscillatie, kruip-herstel en uniaxiale uitrekbaarheid. Tot slot wordt het broodbereidingsproces beknopt besproken en wordt er een samenvatting gegeven van de reologische parameters van deeg waarvan een relatie werd aangetoond met het bakpotentieel van tarwebloem.

De reologische eigenschappen van brooddeeg spelen een belangrijke rol in de verwerking van het deeg en beïnvloeden de broodkwaliteit. Omdat de samenstelling en kwaliteit van tarwebloem zeer variabel is, werden verscheidene empirische reologische methoden ontwikkeld om de functionaliteit van tarwebloem in broodbereiding te gaan voorspellen. Ondanks het feit dat deze methoden hun nut hebben bewezen in de standaard kwaliteits-controle in de industrie, is hun toepasbaarheid in onderzoek beperkt. Daarom werd in dit onderzoek, een meer fundamentele aanpak gehanteerd door het gebruik van rotationele reometrie voor het karakteriseren van de deegreologie.

In deze thesis, wordt de uitdrukking ‘rotationele reometrie’ gebruikt als een gemeenschappelijke term voor twee methoden die worden uitgevoerd op een rotationele reometer, namelijk dynamische oscillatie en kruip-herstel metingen. In dynamisch oscillatie, wordt een deegstaal onderworpen aan een kleine, niet destructieve vervorming die toelaat de lineaire visco-elastische eigenschappen van het deeg te bepalen. Ondanks het feit dat het gebruik van dynamische oscillatie metingen wijdverspreid is voor de karakterisatie van deegsystemen, ontbreekt tot nog toe een duidelijke relatie met bakkwaliteit.
In kruip-herstel metingen, wordt gebruik gemaakt van een constante afschuifspanning die een vervorming van het deegstaal veroorzaakt in functie van de tijd. Wanneer de afschuifspanning wordt verwijderd, kan het elastisch herstel van het staal opgemeten worden. Het mogelijke voordeel van kruip-herstel metingen ten opzichte van de dynamische metingen is dat afschuifspanningen kunnen worden toegepast die beter aansluiten bij de werkelijke verwerkingsomstandigheden van het deeg en aldus kunnen de niet-lineare visco-elastische eigenschappen van het deeg worden bepaald.

Bij het begin van dit onderzoek werd een grondige optimalisatie uitgevoerd van de methode voor het analyseren van de reologische eigenschappen van deeg. Dit was relevant omdat brooddeeg erg gevoelig is voor manipulatie. Vervolgens werd ook het protocol voor de kruip-herstel metingen ontwikkeld. De veranderingen in reologische eigenschappen van deeg ten gevolge van duur van de kruipfase (5-10-15-20 min) en de grootte van de opgelegde afschuifspanning (10-100-250-500-1000 Pa) werden onderzocht. Het Burgers model werd geselecteerd om de data te modelleren. Er werd vastgesteld dat een hersteltijd van 10 minuten voldoende was om het merendeel van het herstel op te meten. Een kruipfase van 5 min bleek reeds voldoende om (pseudo) steady state condities te bereiken. Er werd ook aangetoond dat de grootte van de afschuifspanning een sterke invloed had op de kruipvervorming, maar het daaropvolgende herstel minder beïnvloedde. Vooral de retardatietijden van het herstel waren gevoelig voor veranderingen in de toegepaste afschuifspanning.

De geoptimaliseerde methoden werden vervolgens toegepast voor het bepalen van de reologische eigenschappen van de bloem-water degen van 17 tarwe variëteiten. De tarwebloem werd eveneens geanalyseerd voor de standaard kwaliteitsparameters, farinograaf en alveograaf empirische eigenschappen en zijn bakpotentieel, bepaald als het bekomen broodvolume in een standaard baktest. Dit liet toe om de resultaten met betrekking tot de rotationele reometrie te linken aan de standaard kwaliteitsparameters en het bakpotentieel van

tarwebloem. De belangrijkste conclusie van deze studie was dat de parameters bekomen via rotationele reometrie, meer bepaald de fasehoek (δ) en de dynamische moduli, het beste gerelateerd waren met broodvolume. Algemeen kan worden gesteld dat de reologische eigenschappen van het bloem-water deeg opgemeten door dynamische oscillatie of kruip-herstel, gerelateerd zijn aan de mogelijkheid van het brooddeeg om expansie te vertonen tijdens de broodbereiding.
Verder werd met behulp van principale componenten analyse vastgesteld dat het mogelijk was om de tarwevariëteiten in drie groepen in te delen volgens gemeenschappelijke eigenschappen bekomen uit de kruip-herstel metingen. De indeling was gebaseerd op het vervorminggedrag van het deeg tijdens de kruip-herstel metingen en de snelheid van het elastisch herstel gedurende de herstelfase.

Omdat kneden een belangrijke stap is in de ontwikkeling van de gewenste deegstructuur, werd de invloed van het kneedproces op de reologie en microstructuur van deeg in detail onderzocht. Hiervoor werd gebruik gemaakt van twee modeldeegsystemen, een eenvoudig bloem-water deeg en een bakformulatie deeg, waaraan ook zout en ascorbinezuur werden toegevoegd zoals in de broodbereiding. De twee model deegsystemen werden gekneed gedurende verschillende kneedtijden in twee verschillende kneders, meer bepaald een pin-kneder en een z-arm kneder. Naast rotationele reometrie, werden ook uniaxiale uitrekbaarheidstesten op het deeg uitgevoerd. Significante veranderingen in de reologie en microstructuur van deeg werden waargenomen ten gevolge van het kneedproces. Algemeen kan worden gesteld dat rotationele reometrie de gevoeligste methode was voor het observeren van veranderingen in microstructuur tijdens het kneden van het bloem-water deeg terwijl uniaxiale uitrekbaarheid beter de veranderingen in deegsterkte kon aantonen van het bakformulatie deeg. Voor de bloem-water degen werden weinig verschillen waargenomen tussen degen gekneed in beide kneedtypes. Voor de bakformulatie werd een verschil in deegsterkte opgemerkt gerelateerd aan het type kneder. Via confocale scanning laser microscopie (CSLM), was het mogelijk om verschillen in deegstructuur ten gevolge van kneedtijd, type kneder en deegformulatie te visualiseren. De CSLM beelden boden een verklaring van de reologische observaties. Tot slot werd opgemerkt dat ondanks de verschillen in reologie, kneden voorbij de maximale deegsterkte de broodkwaliteit niet negatief beïnvloedde.

In het laatste deel van het onderzoek werd de invloed van het broodbereidingsproces op de reologie en microstructuur van brooddeeg onderzocht. Vooral de invloed van de verwerkingsstappen zoals opbollen en uitrollen werd in detail bekeken. Hiervoor werd tarwebloem van twee tarwevariëteiten verschillend in kwaliteit geselecteerd. Van deze tarwebloem werden brooddegen zonder gist aangemaakt en verwerkt volgens de standaard bakprocedure. Reologische parameters bekomen via rotationele reometrie waren gevoelig voor veranderingen in deegeigenschappen ten gevolge van de deegverwerking. CSLM toonde aan dat de microstructuur sterk werd beïnvloed door het broodbereidingsproces. Vooral uitrollen van het deeg, veroorzaakte grote veranderingen in het glutennetwerk, en het effect was afhankelijk van de tarwebloemkwaliteit. De verschillen in microstructuur konden een verklaring bieden voor de geobserveerde verschillen in bakkwaliteit. Er werd ook vastgesteld dat hoewel de degen gelijkaardige reologische eigenschappen vertoonden na het kneedproces, deegverwerking de microstructuur op dusdanige wijze kan veranderen dat dit leidt tot een verschil in bakkwaliteit.

Summary

Among cereals, only wheat is capable of forming a dough which has the viscoelastic properties required in breadmaking. A wheat flour dough shows a unique balance between viscous properties, which allow expansion of the dough during fermentation, and elastic properties, which deliver strength to the gas cell membranes in the dough. The aim of this research was to gain more insight in the microstructure and rheology of wheat flour dough influenced by wheat flour quality and dough processing conditions, more precisely during mixing and breadmaking.

In the literature review a general introduction on wheat flour composition, dough formation and dough microstructure is presented. An overview is included on experimental techniques used to study dough rheology followed by the theoretical background of the rheological methods used in this research, more precisely dynamic oscillation, creep-recovery and uniaxial extension. The literature review is concluded with a short overview of the breadmaking process and a summary of rheological parameters which have been shown to relate to the baking potential of wheat flour.

The rheological properties of a bread dough play an important role in dough processing and influence bread quality. Because wheat flour is highly variable in composition and quality, many empirical rheological methods have been developed to measure dough rheology and to predict wheat flour performance in breadmaking. Although these methods have been shown to be useful in standard quality control in industry, their applicability in research is limited. For this reason, a more fundamental approach was followed in this research by applying rotational rheometry to characterize dough rheology.

In this thesis, the expression ‘rotational rheometry’ is used as a common term for the two methods which are performed on the rotational rheometer, namely dynamic oscillation and creep-recovery. In dynamic oscillation a dough sample is subjected to small deformations

which do not damage the dough structure and allow to determine the linear viscoelastic properties. Although dynamic oscillation is widely used for dough systems, no clear relation with baking quality has been reported.
In creep-recovery on the other hand, a constant shear stress is applied and the deformation of the sample is followed as function of time. When the stress is removed, the elastic recovery of the sample is recorded. The possible advantage of creep-recovery compared to dynamic oscillation is that shear stresses may be applied which are closer to the actual processing conditions and thus the non-linear viscoelastic properties can be determined.

At the start of this research a thorough investigation of the effect of sample loading on the rheological parameters was carried out. This is relevant because bread dough is very sensitive to manipulation. Furthermore, the creep-recovery protocol was developed. The changes in dough viscoelastic properties due to varying creep time (5-10-15-20 min) and shear stress (10-100-250-500-1000 Pa) were investigated. The Burgers model was found useful to describe the creep-recovery data. Further, a recovery time of 10 minutes was found to be sufficient to obtain most of the recovery after the creep deformation. It was also observed that a creep time of 5 minutes was sufficient to reach (pseudo) steady state conditions in creep. Shear stress was shown to strongly influence the creep response but total recovery was less harmed. The recovery retardation times, on the other hand, were very sensitive to changes in shear stress.

The optimized methods were then used to analyze the rheological properties of flour-water doughs of wheat flour obtained from 17 pure wheat cultivars. The wheat flour was also analyzed for a set of standard quality parameters, farinograph and alveograph empirical rheological properties and breadmaking potential. This allowed to compare the results of rotational rheometry with the standard quality tests and baking volume. The major outcome of this study was that from all the obtained parameters and in contrast to the existing literature, phase angle delta and the dynamic moduli were the parameters best related to bread volume. In general, the rheological properties of flour-water doughs as measured by rotational rheometry, were found to be related to the ability of the bread dough to expand during breadmaking.
Secondly, through principal component analysis, it was possible to divide the wheat cultivars into three groups with similar rheological behaviour based on their overall deformability in creep-recovery and the recovery retardation time (r_2), which indicates how fast elastic recovery occurs after deformation.

As mixing is a crucial step in obtaining the desired dough structure, the effect of the mixing process on dough rheology and microstructure was investigated in more detail. For this purpose two model dough systems were used, more precisely a simple flour-water dough and a flour-water dough to which also ascorbic acid and salt were added as a simulation of the breadmaking dough. The two dough formulations were mixed for different times in two dough mixers which are believed to exert a different mixing action, namely a pin mixer and a z-blade mixer. Next to rotational rheometry, also uniaxial extension properties were determined. Significant changes in dough rheology and dough microstructure were observed as result of the mixing process. In general, rotational rheometry was sensitive for changes in microstructure of the flour-water dough during mixing, whereas uniaxial extension reflected best the changes in dough strength of the baking formulation dough upon mixing. Differences in dough structure development and dough strength were observed due to mixer type. CSLM revealed differences in dough microstructure caused by mixing time, mixer type and dough formulation which may explain the observed changes in dough rheology. However the mixing process caused relevant changes to dough rheology and microstructure, is was observed that mixing past maximum dough strength did not negatively affect baking quality.

In the last part of the experimental work, the effect of the breadmaking process on dough rheology and microstructure was studied. Especially the influence of the processing steps (rounding and moulding) was of interest. For this purpose, bread doughs prepared from two wheat flours differing in quality were prepared with all baking ingredients except yeast and processed according to the standard baking test procedure. Rheological parameters obtained from rotational rheometry showed to be sensitive to changes in dough properties caused by processing. CSLM images showed that the processing steps in the breadmaking procedure caused major changes in the microstructure of the bread dough. Especially moulding had a large effect on microstructure, depending on the flour type. The differences in microstructure may offer an explanation for the observed differences in baking quality. It was found that although doughs may have similar rheological properties and microstructure after mixing, dough processing may affect microstructure in a different way leading to differences in baking quality.

Outline of the research

Among cereals, only wheat is capable of forming a dough which has the viscoelastic properties required in breadmaking. A wheat flour dough has a unique balance between viscous properties, which allow expansion of the dough during fermentation, and elastic properties, which deliver strength to the gas cell membranes and thus prevent collapse of the dough structure. The aim of this research was to gain more insight in the microstructure and rheology of wheat flour dough systems and to elucidate the influence of wheat flour quality and dough processing conditions during mixing and breadmaking.

The outline of the research is presented in Figure 1. A general introduction on wheat flour, dough formation and dough microstructure is provided in Chapter 1. In addition, a summary of the rheological methods used to analyze the viscoelastic properties of wheat flour dough and the theoretical background of the rheological methods used in this research are presented.

As several methods in this thesis are of importance for the experimental work described in the different chapters, they are joined and described in Chapter 2.

In Chapter 3, the rheological method for analyzing dough rheological properties by means of rotational rheometry is described. The sample loading protocol and the creep-recovery methodology are presented. This chapter provides the base for the experimental work for the following chapters.

The application of rotational rheometry in the evaluation of the rheological properties of wheat flour is investigated in Chapter 4. Wheat flours obtained from seventeen pure wheat cultivars are analyzed for standard quality parameters and baking potential. In addition, flour-water doughs are prepared and dough viscoelasticity is analyzed by performing dynamic

oscillation and creep-recovery measurements. A thorough discussion on the relationship between standard quality parameters, rheological properties and baking volume, is presented in Chapter 4.

The influence of the mixing process on dough rheology and microstructure is investigated in detail in Chapter 5. The effect of mixer type (pin mixer vs. z-blade mixer), mixing time and dough formulation is investigated. Next to rotational rheometry, uniaxial extension testing is applied to study dough rheology. CSLM is used for visualization of the dough microstructure.

In Chapter 6 the changes in dough rheology and microstructure during breadmaking are studied for two wheat flours which differ in baking quality. The effect of dough processing (rounding and moulding) on dough rheology is investigated and dough microstructure is visualized by CSLM.

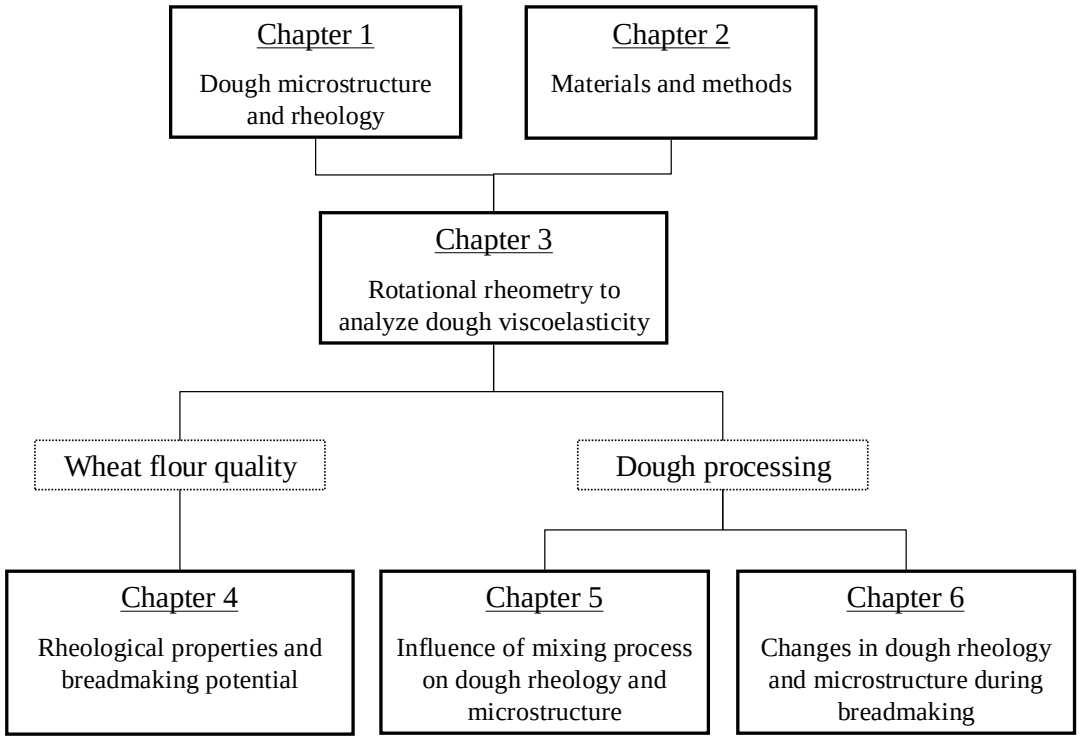

Figure 1 Outline of the research

Chapter 1

Dough microstructure and rheology

BROEINSJEL: van ’t graan: het bloeien.

[Oilsjtersen Diksjoneir]

1.1. Introduction

Bread has been produced for thousands of years and today it is still an essential part of the human diet. Although breads in different parts of the world vary largely in composition, shape, taste and texture, their main ingredient is usually the same, namely wheat flour. When wheat flour and water are combined and energy is added during mixing, a dough is formed with unique viscoelastic properties. Among all cereals, only wheat flour is capable of producing such a dough system which is able to trap the carbon dioxide released during fermentation resulting in a leavened bread. It is widely accepted that the unique viscoelastic properties of a wheat flour dough may be attributed to the presence of the gluten proteins.

For breadmaking, wheat flour with an appropriate quality is required. However, wheat flour varies largely in quality determined by wheat cultivar, growing location and cultivation conditions. In industrial milling practice wheat flours of different quality are blended to obtain wheat flour with the required properties depending on the application. To determine wheat flour quality, many test procedures have been developed among which several rheological methods to characterize dough viscoelastic properties. Dough rheological properties are important as they affect the processing properties of the dough and also influence the final bread quality.

In this chapter a general introduction is given to wheat flour, dough microstructure and dough viscoelasticity. Further, the methods which are commonly applied to measure dough rheology are summarised and the theoretical background of the rheological methods used in this research is presented in more detail.

1.2. Bread dough microstructure

1.2.1. Wheat flour

1.2.1.1. Origin

Wheat belongs to the family of the grasses (*Poaceae*, syn. *Gramineae*) and is among the oldest and most extensively grown of all grain crops. In 2009, wheat was the second most produced cereal worldwide with an annual production volume around 680 million tonnes

(FAOSTAT 2011). The term wheat describes a number of species and subspecies in the genus *Triticum*, but today the most important are bread wheat (*T. aestivum* subsp. *aestivum*) and durum wheat (*T. turgidum* subsp. *durum*), accounting for 90 and 5% of total world wheat production respectively (Gooding, 2009).

Wheat flour is obtained after milling of the wheat grains. The wheat grain or wheat kernel consists of the germ and the starchy endosperm which are enclosed by the bran layers (Figure 1.1). In the milling process the germ and bran are separated from the endosperm of which the particle size is then further reduced to obtain a white flour. The distribution of components in the grain determines whether they are present in the white flour produced by milling (Bechtel et al., 2009). Flour composition will also differ along different flour streams during milling and will be influenced by the extraction rate (Eliasson and Larsson, 1993).

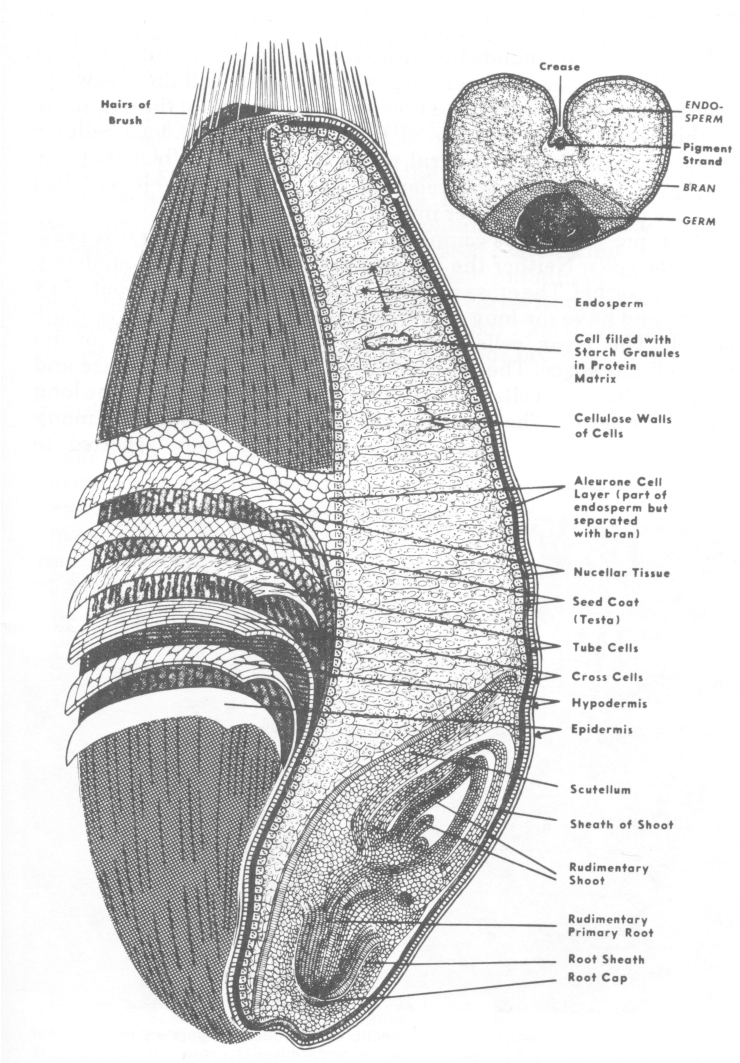

Figure 1.1 Longitudinal and cross section of a wheat kernel (Hoseney, 1992)

1.2.1.2. Composition

Based on dry matter, wheat flour mostly contains starch (70-75%), proteins (8-18%), lipids (ca. 1-2%) and non-starch polysaccharides (2-3%).

Starch is present in wheat flour as semi-crystalline granules which are composed of amylose and amylopectin. There are two main classes of starch granules. The larger 'A' granules have lenticular shape and a diameter from 15 to 30 µm whereas 'B' granules have a more spherical structure and a diameter around 10 µm (Stone and Morell, 2009).

Wheat proteins are a highly heterogeneous mixture of proteins which are not easily separated and quantified. Osborne (1907) showed that wheat proteins may be separated into four fractions based on their differences in solubility: albumins which are soluble in water, globulins which are soluble in salt water, gliadins which are soluble in 70% ethanol and glutenins, partly soluble in dilute acid or alkali. The gliadins and glutenins, which form the gluten, are also called the storage proteins and represent ca. 80% of the total wheat protein in a typical wheat flour. Gliadins are a heterogeneous mixture of single-chained polypeptides with a molecular weight range of ca. 30000 to 75000 Da. According to their electrophoretic mobility they are divided into four groups: α-, β-, γ-, and ω-gliadins (Gianibelli et al., 2001). The glutenin fraction comprises high molecular weight glutenin (HMW) and low molecular weight (LMW) subunits, which are linked by interchain disulfide bonds to form polymers ranging in size from dimers to components with molecular weights in the millions (Shewry et al., 2009).

The gluten proteins have unique properties as they can form a viscoelastic network which gives a dough the ability to retain gas during fermentation and baking. The contributions of gliadins and glutenins to dough viscoelasticity have long been recognized and it has been suggested that gliadins generally contribute to dough viscosity and glutenins contribute to dough elasticity and dough strength (Khatkar et al., 1995; Uthayakumaran et al., 2000). It is also generally accepted that the breadmaking quality of a wheat flour is largely determined by the amount and quality of the gluten proteins present. A schematic overview (Figure 1.2) of the effect of protein quantity and quality on dough rheology and breadmaking quality was presented by Goesaert et al. (2005). Extensive literature is available on the gluten protein composition and the functionality of the separate gluten fractions (Anjum et al., 2007; Gianibelli et al., 2001; Shewry et al., 2009; Shewry et al., 2000; Veraverbeke and Delcour, 2002). From the gluten fractions, especially the large insoluble glutenin polymers determine

the dough rheological properties. This fraction is often called the glutenin macro polymer (GMP) which consists out of very large glutenin protein aggregates (Don et al., 2003a).

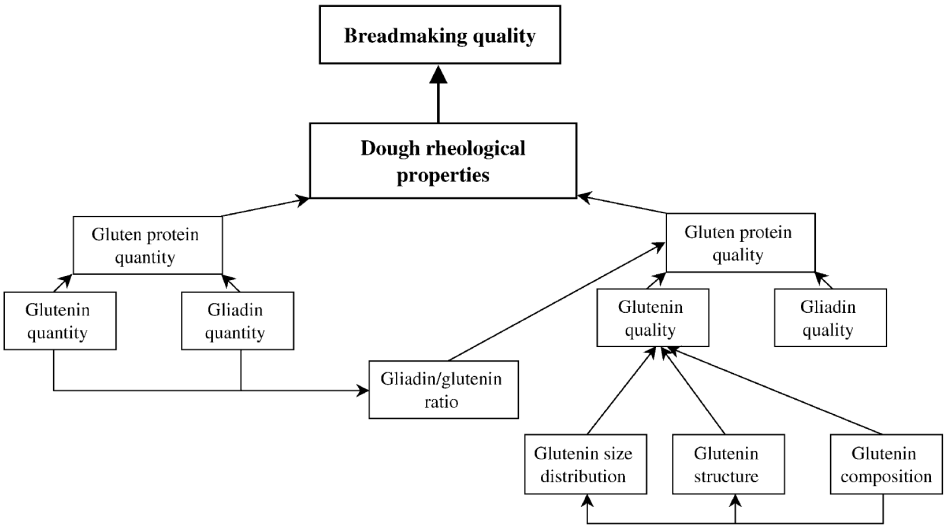

Figure 1.2 Schematic overview of dough rheology as affected by gluten quantity and quality (Goesaert et al., 2005)

Although the rheological properties are largely determined by the wheat gluten proteins, interactions of the gluten protein matrix with other flour components (e.g. flour lipids, arabinoxylans, non-gluten proteins) may affect the rheological properties (Veraverbeke and Delcour, 2002). Wheat lipids and non-starch polysaccharides are minor flour constituents but they may have a large impact on wheat flour functionality. Flour lipids have significant effects on baking performance in terms of volume and crumb structure (MacRitchie and Gras, 1973; Sroan and MacRitchie, 2009). Non-starch polysaccharides, in particular arabinoxylans, have the capacity to significantly affect the properties of the dough and the baked product (Courtin and Delcour, 2002).

1.2.2. Gluten viscoelasticity

The rheological behaviour of wheat flour dough is relevant for several aspects. It may provide the base for understanding the handling properties of the dough and may even predict the quality of the final bread. Furthermore, rheology provides the possibility to relate macroscopic properties to dough microstructure (Eliasson and Larsson, 1993). As mentioned before, the gluten proteins are the main responsible for the unique viscoelastic properties of a

dough. To explain the viscoelastic properties of a dough, several models based on gluten network structure have been proposed. However, gluten network structure is still not understood completely.

The earliest models for explaining gluten structure were based on the molecular interactions, more specifically the disulfide bonds, between gluten proteins (Bloksma, 1990a; Greenwood and Ewart, 1975). Figure 1.3 summarizes a widely-held view of gluten structure in which the HMW subunits form an 'elastic backbone' consisting largely of head-to-tail polymers with inter-chain disulfide bonds. This backbone forms a base for LMW subunit 'branches' (linked by disulfide bonds). Gliadins may also interact with the glutenin polymers by non-covalent forces, although these interactions are traditionally considered to contribute to gluten viscosity rather than elasticity (Graveland et al., 1985; Shewry et al., 2000).

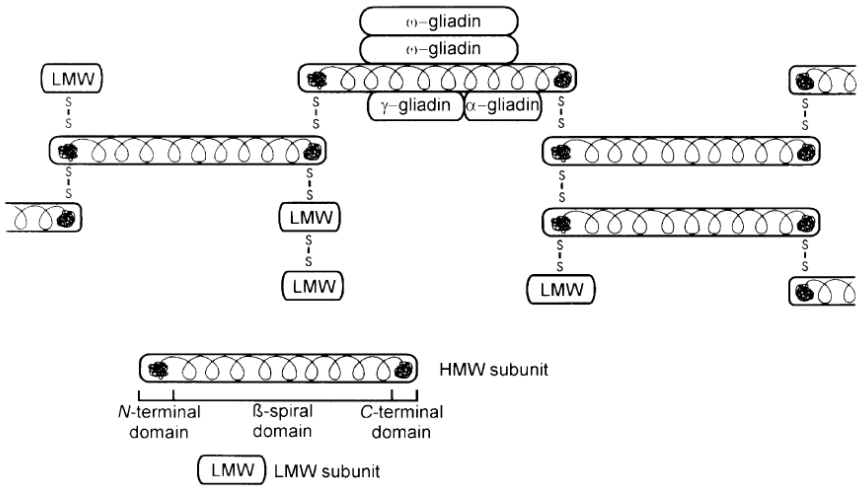

Figure 1.3 A structural model for wheat gluten in which the HMW subunits provide a disulfide-bonded backbone which interacts with other gluten proteins by disulfide bonds (LMW subunits) and non-covalent interactions (gliadins) (Shewry et al., 2000)

As the earlier models could not explain all the dough properties, new models were proposed also taking into account the physical interactions between gluten proteins on the molecular level. Belton (1999) presented a loop and train model to explain the elastic properties of gluten. This model postulates that in gluten, there are regions of the protein chains that are held by interchain hydrogen bonds (trains) as well as unbounded regions (loops). Stretching of gluten first extends the loops and then causes the proteins to slide over one another. The elastic restoring force is then due to the drive to re-establish the loop-train equilibrium of the unstretched polymer (Hamer et al., 2009).

According to Dobraszczyk and Morgenstern (2003), dough behaves like many polymer systems as it is extensible. Their model is based on the presence of entanglements between chain segments. Coiled chains between entanglements are initially stretched, after which further flow depends on slippage of chains through entanglement nodes (Hamer et al., 2009; Singh and MacRitchie, 2001).

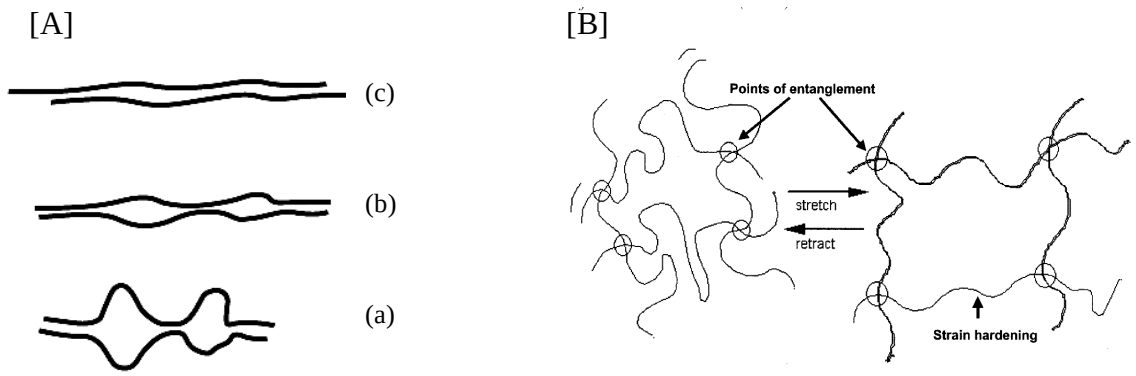

Figure 1.4. [A] The loop and train model as proposed by Belton (1999). Deformation of polymers resulting from extending the network: (a) equilibrium configuration (b) small extension – only the loops are deformed (c) large extension loops are flattened and the interchain hydrogen bonds are broken so that the chains slip over each other. [B] Model of entanglement network in a high MW polymer during stretching (Dobraszczyk and Morgenstern, 2003).

Hamer and van Vliet (2000) proposed a model for glutenin hyper-aggregation, which explains the impact of both physical and chemical interactions (covalent versus non-covalent processes) from the gluten(in) structure at various length scales. This model suggests the presence of mesoscopic glutenin particles (ca. 10-100 μm) as the main building blocks of the gluten(in) network in a dough. The protein particles can be deformed and disrupted to form a protein particle network which is held together by physical interactions (Don et al., 2005). According to this model, the macroscopic scales (100-1000 μm) are solely based on physical (i.e. non-chemical) interactions. It has been proposed that glutenin particle properties are the key element to understand the link between GMP and dough properties (Don et al., 2003b; Don et al., 2005).

It can be concluded that several models for gluten structure have been proposed. However, no agreement has yet been reached on which model is the most suitable for describing gluten

structure and dough viscoelasticity (Belton and Dobraszczyk, 2006; Hamer et al., 2005; van Vliet and Hamer, 2007).

1.2.3. Dough formation

When wheat flour and water are combined and mixing energy is added to the system, a dough will be formed. Flour particles are highly hygroscopic and will quickly absorb water. Flour constituents vary in their water uptake capacity as shown in Table 1.1. When water is added to flour particles, proteinaceous fibrils will appear, which on their turn interact to form a cohesive dough (Amend and Belitz, 1990). Mixing helps to hydrate the flour particles by exposing new surfaces for interaction with water. As mixing proceeds, flour particles lose their identity and the dough takes on a relatively homogeneous appearance (Bushuk, 1998).

Table 1.1 Proximate distribution of water in dough (Bushuk, 1998)

Constituent	%db	WA (g/g)	WD (%)
Starch undamaged	56	0.3	18.9
damaged	24	1.0	27.0
Protein	14	2.0	31.5
Pentosan	2	10.0	22.5

db = dry basis; WA: water absorption capacity; WD: water distribution in dough

During mixing, complex chemical and physical interactions take place between the different constituents in the dough system (Bloksma and Bushuk, 1988). Shear and extensional stresses imparted by mixing cause dispersion of gluten throughout the dough (Peighambardoust et al., 2006). During mixing and subsequent resting, de-polymerization and re-polymerisation of glutenin occurs (Weegels et al., 1997). Mixing changes the solubility of the glutenin polymers by decreasing their polymer size, which renders them more soluble. At peak dough development the amount of glutenin macropolymer (GMP), which is a measure of the insoluble highly polymerized glutenin fraction, was found to be almost zero (Don et al., 2003b). This indicates important changes in the structure of glutenin polymers during dough formation.

Both covalent and non-covalent bonds are involved during dough formation and in the final dough structure. The disulfide bonds of flour proteins play a key role in the interactions in doughs. They form relatively strong crosslinks within and between polypeptide chains and

also stabilize other less energetic bonds such as hydrogen bonds and hydrophobic interactions (Bushuk, 1998). Next to disulfide bonds, also dityrosine bonds may contribute to dough structure (Tilley et al., 2001). However, during dough formation, the number of crosslinks formed between tyrosine residues appears to be small and of little importance in the structure of the gluten network (Peña et al., 2006).

Other important interactions in dough involve the non-covalent bonds like hydrogen bonds, hydrophobic interactions, ionic bonds and van der Waals bonds. Hydrogen bonds are much weaker than covalent bonds, but because of the large numbers that act cooperatively, they contribute significantly to the structure of the dough (Bushuk, 1998).
The hyper-aggregation model proposed by Hamer and van Vliet (2000) is based on the presence of non-covalent interactions which are dominant at larger length scales. According to this model, disulfide bonds between glutenin subunits are dominant at small length scales (<1µm), while physical interactions are dominant at larger length scales (1-100 µm). The formed particle network is held together by physical interactions (Don et al., 2005). Interactions with non-protein constituents become relevant at even larger length scales (>100 µm).

1.2.4. Dough microstructure

Several models for dough structure have been proposed. According to Aibara et al. (2005) a dough system can be characterized on different scales (Figure 1.5). On a macroscopic level, a dough can be divided into a homogeneous dough phase and a dispersed gas phase. On a microscopic level, the continuous dough phase consists of a continuous gluten phase in which starch and yeast cells are dispersed. On the molecular level, a continuous water phase is observed next to the insoluble gluten proteins. The water phase contains electrolytes, soluble proteins and other soluble components like sugars.

Another model for dough structure was presented by Eliasson and Larsson (1993). They postulated that during mixing a bicontinuous network of two water-containing phases is formed, more precisely the gluten phase and the “free” water phase. Starch granules are present in the “free” water phase and because their surface is covered with water, they will tend to fuse into a continuous network.

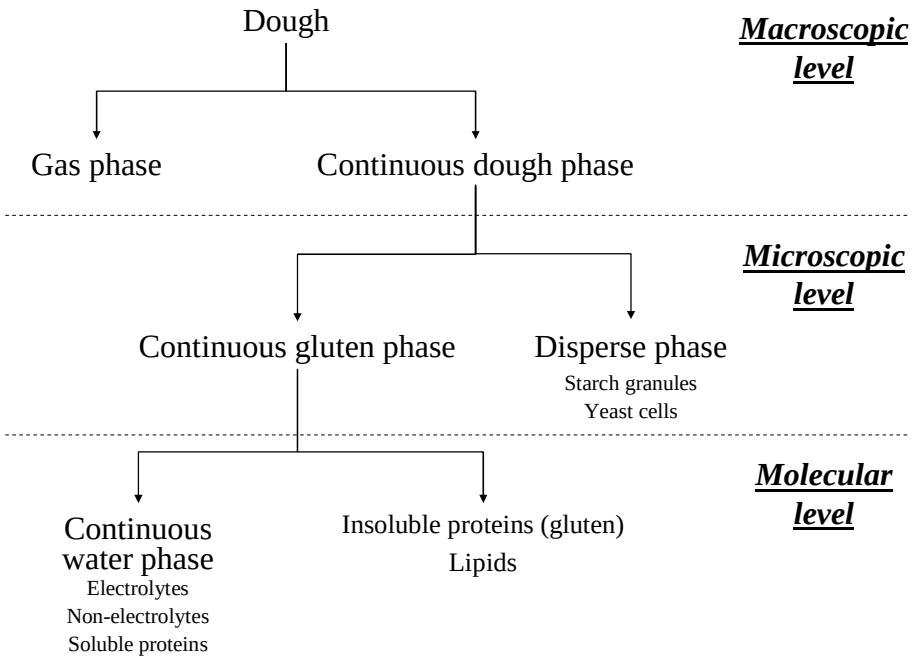

Figure 1.5 Dough structure on different length scales (adapted from Aibara et al., 2005)

Gan et al. (1995) and MacRitchie (2003), on the other hand, suggested that the gas cells in the dough matrix are surrounded by continuous liquid lamellae (Figure 1.6). It is thought that the liquid lamellae surrounding expanding gas cells act as a secondary protection together with the primary gluten-starch matrix, thus preventing coalescence and disproportionation. The stability of the liquid lamellae depends on the absorption of surface active compounds at the liquid-gas interface (Sroan and MacRitchie, 2009).

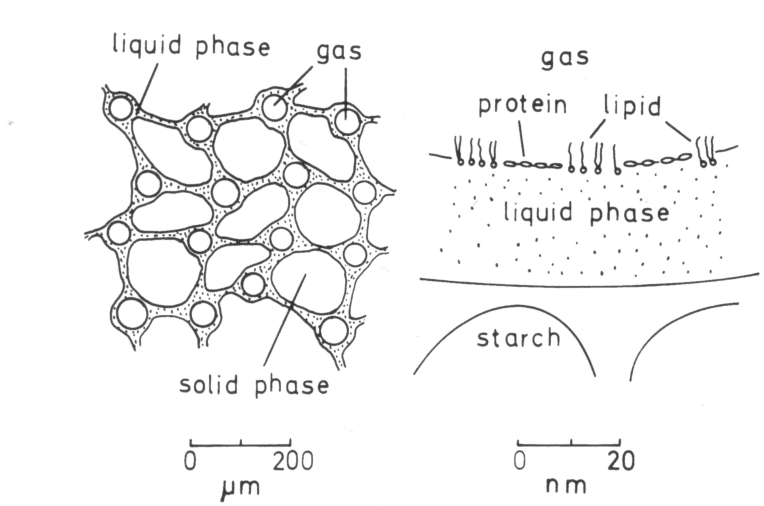

Figure 1.6 Schematic diagrams of dough at two magnifications (MacRitchie, 2003)

The presence of a three-dimensional gluten network has been visualized by scanning electron microscopy (Amend and Belitz, 1990) in which the formation of gluten strands and gluten films during dough development were clearly shown. Peighambardoust et al. (2006) reported that dough microstructure is not only influenced by the amount of energy input but also by the type of the applied deformation. Z-blade mixing involving both shear and elongational deformation, led to a dispersion of the gluten structures and a homogeneous dough. Simple shearing in a cone and plate shearing device (Peighambardoust et al., 2004) on the other hand led to aggregated protein structures in the dough.

1.3. Experimental techniques to measure dough rheology

1.3.1. Introduction

Rheology is the study of flow and deformation of materials and describes how a material responds to an applied stress or strain (Steffe, 1996). The term rheology was introduced by Eugen C. Bingham in 1929 and originates from the Greek “rheos” which means “flow”. However, rheological measurements do not merely reveal information about the flow behaviour of liquids, but also about the deformation behaviour of solids as large deformations produced by shear forces cause many (solid) materials to flow (Mezger, 2002).

In food industry, rheological data are needed in numerous areas such as calculations in process engineering, determination of ingredient functionality in product development, intermediate or final product quality control, shelf life testing, sensorial analysis and construction of rheological models (Steffe, 1996).

Materials may be divided according to their rheological behaviour which may be viscous, elastic or viscoelastic. Materials showing viscous behaviour will deform at a certain rate as soon as a stress is applied and the deformation will remain unchanged upon removal of the stress. Elastic materials will deform almost immediately when stress is applied and the original state is regained when the stress is removed. Viscoelastic materials show both viscous and elastic properties when deformed.

Rheological techniques are generally divided into fundamental and empirical methods. Fundamental rheological properties are independent of the technique used whereas empirical rheological properties depend on the measuring equipment. Empirical rheological tests are often used in practical factory settings as they are robust and easy to perform. However, they do not fulfil the requirements of a fundamental rheological test (Dobraszczyk and Morgenstern, 2003), since:

- the sample geometry is variable and not well defined
- the stress and strain rates are uncontrolled, complex and non-uniform
- it is therefore impossible to define any rheological parameter such as stress, strain, strain rate, modulus or viscosity.

Bread dough is a viscoelastic material with explicit nonlinear behaviour. It exhibits shear thinning and thixotropy (Weipert, 1990). Many specific instruments and methods have been developed during the past century to analyze dough viscoelastic properties. An extensive overview of rheological methods used in cereal science is given by Dobraszczyk and Morgenstern (2003). It is beyond the scope of this chapter to review all methods used in dough rheology research. Only the theoretical background of the methods used in this thesis will be discussed in more detail.

1.3.2. Empirical methods

For no other food product more empirical rheological methods have been developed, as for wheat flour dough. Table 1.2 gives an overview of the most common empirical methods used to study dough rheology and the dough properties which are obtained. The methods can be divided into the recording mixers, extension instruments and the fermentometers. The empirical methods are an essential part of wheat flour quality control as several methods have been adopted in the AACC, ICC or ISO international standard methods.

Table 1.2 Overview of the most commonly used empirical methods to measure dough rheology

Method	Company	International standard	Measured properties
Recording mixers			
Farinograph	Brabender	AACC 54-21 ICC 115/1 ISO 5530-1	Water absorption Dough development time Dough resistance (torque) Starch gelatinisation (only DoughLAB and Mixolab)
Mixograph	National manufacturing	AACC 54-40	
Consistograph	Chopin Technologies	AACC 54-50 ICC 117	
DoughLAB	Newport Scientific	-	
Mixolab	Chopin Technologies	AACC 54-60 ICC 173	
Extension instruments			
Extensograph	Brabender	AACC 54-10 ICC 114/1 ISO 5530-2	Dough extensibility Resistance to extension Deformation energy
Kieffer extensibility rig	Stable Micro Systems	-	
Alveograph	Chopin Technologies	AACC 54-30 ICC 121 ISO 21971	
Dough inflation system	Stable Micro Systems	-	
Fermentometers			
Rheofermentometer	Chopin Technologies	AACC 89-01	Yeast activity Dough volume Gas retention
Fermentograph	Brabender	-	

1.3.3. Fundamental methods

Fundamental rheological methods can be divided according to the type of deformation applied on the sample (e.g. compression, extension or shear) and also according to the relative magnitude of the imposed deformation: e.g. small or large deformation (Dobraszczyk and Morgenstern, 2003). Fundamental methods used to study dough rheology are listed in Table 1.3.

The theoretical background of rotational rheometry and uniaxial extension will be given in more detail. For rotational rheometry, two methods will be discussed, more precisely dynamic oscillation and creep-recovery.

Table 1.3 Overview of fundamental methods to measure dough rheology (based on Dobraszczyk and Morgenstern, 2003)

Method	Deformation type	Deformation magnitude	Geometry	Property measured
Dynamic oscillation	shear	small	parallel plates	dynamic moduli phase angle delta
Transient flow	shear	small/large	parallel plates	creep-recovery stress relaxation
Uniaxial extension	extension	large	Kieffer rig extensional rheometry	fracture properties strain hardening extensional viscosity
Biaxial extension	extension	large	dough inflation system Alveograph	fracture properties strain hardening extensional viscosity
Biaxial extension	compression	large	parallel plates	fracture properties strain hardening extensional viscosity

1.3.3.1. Rotational rheometry

A rotational rheometer is the most frequently used type of equipment to determine the fundamental rheological properties of foods and many other materials and involves the shearing of the test materials between rotating cylinders, cones or plates (Whorlow, 1992). In comparison with other types of rheological equipment, rotational rheometers offer the advantage that the sample can be sheared for as long as desired, allowing the study of time-dependency. At the appropriate conditions the entire sample can be subjected to a uniform shear rate (Verbeken, 2006).

When a shear stress σ (i.e. shear force divided by the area over which the force is distributed, [Pa]) is applied to a material, it will deform as is shown in Figure 1.7. The deformation or strain can be expressed as:

$$\gamma = \frac{du}{h} = tg\alpha \qquad \textbf{(1-1)}$$

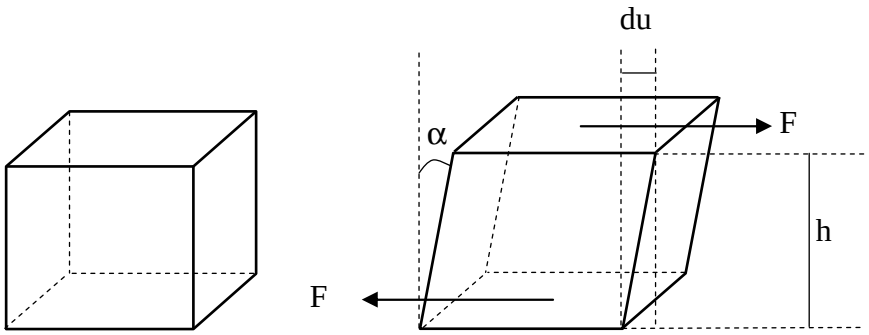

Figure 1.7 Deformation caused by a shear force

For an ideal or Hookean solid, the resulting strain is proportional to the applied shear stress as is expressed by Hooke's law:

$$\sigma = G \times \gamma \quad \textbf{(1-2)}$$

where G is the shear modulus or modulus of rigidity [Pa]. When the shear stress is removed, an ideal solid will return to its initial shape and size, which is called 'elastic behaviour'.

An ideal or Newtonian liquid subjected to a shear stress will continue to deform as long as the stress is applied. The deformation rate or shear rate [s^{-1}] is given by:

$$\dot{\gamma} = d\gamma / dt \quad \textbf{(1-3)}$$

For a Newtonian liquid, the shear rate is proportional to the magnitude of the applied stress:

$$\sigma = \eta \times \dot{\gamma} \quad \textbf{(1-4)}$$

where η is the viscosity [Pa.s]. Upon removal of the stress the material will not recover from its deformation, which is called 'viscous behaviour'.

Ideal elastic and ideal viscous behaviours present two extreme responses of materials to external stresses. Real materials, however, exhibit a wide array of responses between viscous and elastic. Most materials exhibit some viscous and some elastic behaviour simultaneously and are called 'viscoelastic'. The viscoelastic properties of materials are determined by dynamic or transient methods (Gunasekaran and Ak, 2000).

1.3.3.1.1 Dynamic oscillation

Dynamic or oscillatory tests are widely used to study the viscoelastic behaviour of food. In this type of test procedures the sample is subjected to small amplitude sinusoidal strains and the resulting stress is recorded. These small amplitude oscillatory tests are commonly performed in shear and hence abbreviated as SAOS, as for small amplitude oscillatory shear (Gunasekaran and Ak, 2000). The material deformation during an oscillatory test is shown in Figure 1.8. This method is very sensitive to chemical composition and physical structure what makes it very useful in a wide range of applications (Steffe, 1996).

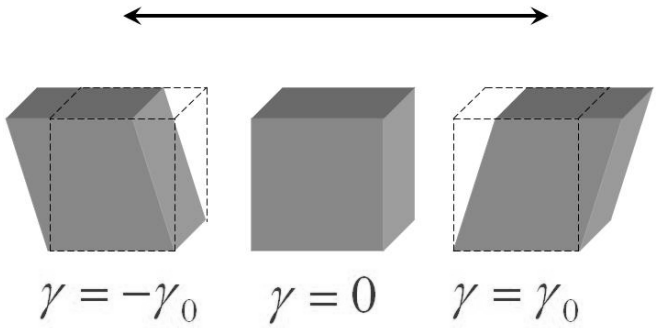

Figure 1.8 Material deformation during an oscillatory test

Dynamic testing instruments may be divided into two categories: controlled rate instruments where the deformation (strain) is fixed and stress is measured, and controlled stress instruments where the stress amplitude is fixed and the deformation is measured. For a controlled rate instrument the strain as a function of time is given by:

$$\gamma = \gamma_0 \sin(\varpi t) \quad \textbf{(1-5)}$$

with γ_0 the maximum deformation amplitude and ω [rad/s] the oscillatory frequency. The sinusoidal strain input results in a periodic shear rate:

$$\dot{\gamma} = \frac{d\gamma}{dt} = \gamma_0 \varpi \cos(\varpi t) \quad \textbf{(1-6)}$$

The stress [Pa] produced by the strain input is given by:

$$\sigma = \sigma_0 \sin(\varpi t + \delta) \quad \textbf{(1-7)}$$

with σ_0 being the maximum stress or stress amplitude and δ [°] the phase shift relative to the applied strain, also referred to as phase angle.

In an ideal solid, the stress will be in phase with the strain (δ=0°) as all the energy will be stored. For an ideal viscous material on the other hand, the stress will be 90° out of phase as all the energy is dissipated (Whorlow, 1992). A viscoelastic material will show a phase shift between 0° and 90° depending on the relative elastic and viscous component of the material (Figure 1.9).

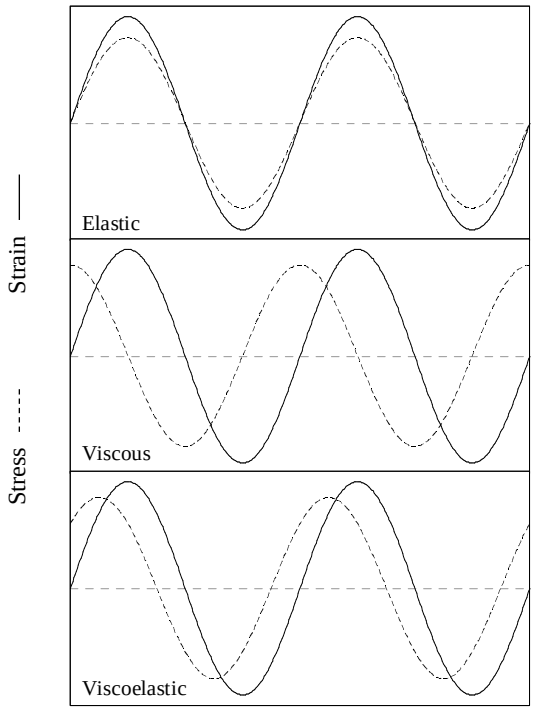

Figure 1.9 Dynamic response of an elastic, viscous and viscoelastic material to oscillatory shear (Based on Tung and Paulson, 1995)

Based on trigonometry, the stress function (1-7) can be split up into two components:

$$\sigma = \sigma_0 \cos\delta \sin(\varpi t) \quad + \quad \sigma_0 \sin\delta \cos(\varpi t) \qquad \textbf{(1-8)}$$

with the first component being in phase and the second component out of phase (90°) with the applied strain. When taking into account equations (1-5) and (1-6), the stress function (1-8) can be rewritten as:

$$\sigma = G' \gamma + (G''/\varpi)\, \dot{\gamma} \qquad \textbf{(1-9)}$$

in which G' and G" are function of the amplitude ratio and the phase shift:

$$G' = \frac{\sigma_0}{\gamma_0} \cos\delta \quad \textbf{(1-10)}$$

$$G'' = \frac{\sigma_0}{\gamma_0} \sin\delta \quad \textbf{(1-11)}$$

The storage or elastic modulus G' [Pa] is a measure of the energy that is stored and recovered per oscillation cycle. The loss or viscous modulus G" [Pa] is an estimate of the energy that is dissipated as heat per oscillation cycle. Both moduli are function of frequency.

The ratio of the loss and storage modulus is equal to the tangent of the phase angle, also referred to as the loss tangent:

$$\tan\delta = \frac{G''}{G'} \quad \textbf{(1-12)}$$

Finally, the complex modulus G* combines the in and out of phase component and can be written as

$$G^* = G' + iG'' \quad \textbf{(1-13)}$$

The modulus of the complex modulus |G*| [Pa] is defined as the ratio of the stress amplitude to the strain amplitude:

$$\left|G^*\right| = \frac{\sigma_0}{\gamma_0} = \sqrt{(G')^2 + (G'')^2} \quad \textbf{(1-14)}$$

The modulus of the complex modulus |G*| is regarded as a measure of the consistency of a material and is often called the complex modulus in practice.

Dynamic oscillation tests can be performed in different modes. In an amplitude sweep a sample is subjected to an increasing stress or strain while the frequency is kept constant. Amplitude sweeps are generally used to determine the linear viscoelastic region (LVR) in which stress and strain are linearly proportional to each other. As can be seen in Figure 1.10, G' and G" remain constant in the LVR.

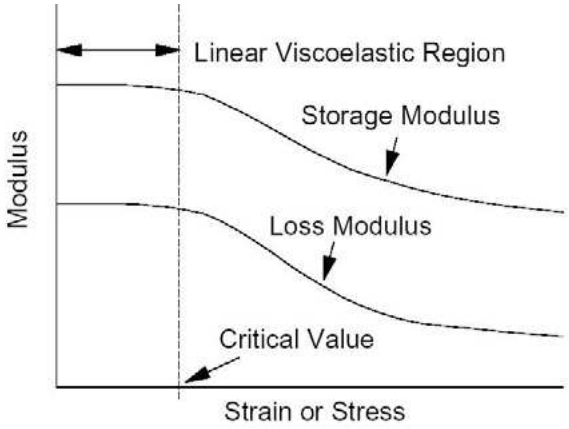

Figure 1.10 Typical response to a strain or stress sweep showing the linear viscoelastic region (Steffe, 1996)

The most common mode of oscillatory testing is probably the frequency sweep because it shows how the viscous and elastic behaviour of the material change with the rate of application (frequency) of strain or stress (Steffe, 1996). In a frequency sweep the stress or strain amplitude is kept constant but the frequency increases. This test type is very useful for comparing different food products or for comparing the effects of various ingredients and processing treatments on viscoelasticity (Steffe, 1996).

An isothermal time sweep, where frequency and amplitude are constant over time, can be used to monitor time-dependent structural changes as firming. When combined with a controlled change in temperature, temperature induced changes can be studied e.g. gelation or crystallization (De Graef, 2009; Verbeken, 2006).

1.3.3.1.2 Creep-recovery

Creep-recovery is a transient test method for viscoelasticity in which an instantaneous stress is applied to a sample and the change in strain (creep) is observed over time. When the stress is released, some recovery may be observed as the material attempts a return to the original shape (Steffe, 1996). Creep-recovery measurements can be performed in compression, uniaxial tension or shear, of which the latter is the most common.

Idealized creep-recovery curves are shown in Figure 1.11. As an ideal elastic material is subjected to a constant stress, it deforms immediately and the strain is constant during the entire creep phase. When the stress is removed, an ideal elastic material will recover instantaneously to its original shape. An ideal viscous material shows steady flow, producing a linear response to stress with the inability to recover any of the imposed deformation. Viscoelastic materials exhibit a non-linear response when subjected to a constant stress. Due to their ability to recover some structure by storing energy, a viscoelastic material shows a permanent deformation less than the total deformation applied to the sample (Steffe, 1996).

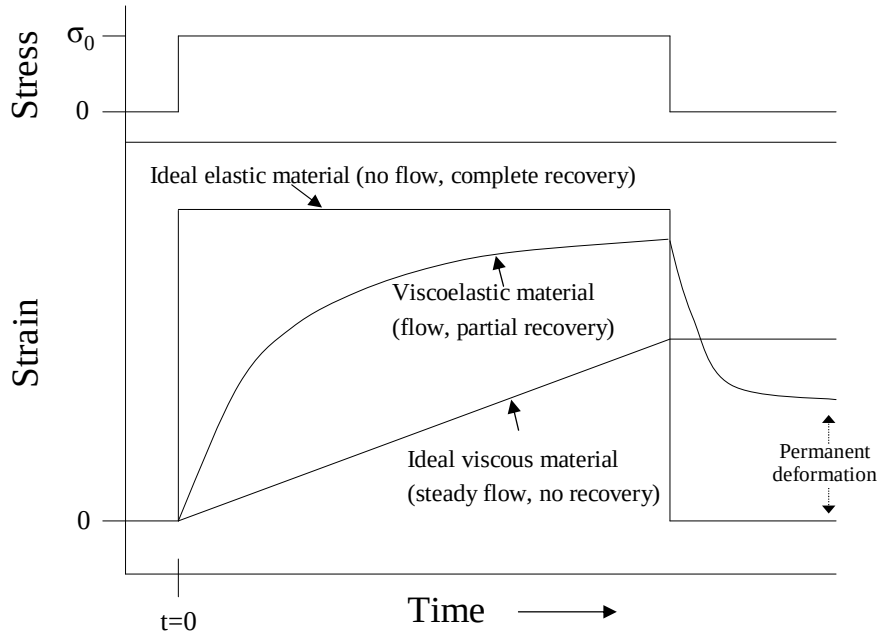

Figure 1.11 Creep and recovery curves of an ideal elastic, an ideal viscous and a viscoelastic material (Adapted from Steffe, 1996)

Creep data are generally described in terms of compliance *J* [1/Pa]:

$$J = f(t) = \frac{\gamma}{\sigma_{cons\tan t}} \qquad \textbf{(1-15)}$$

When data are collected in the LVR, the compliance curves at different applied stresses overlap, indicating that the obtained strain is proportional to the imposed stress.

To investigate creep-recovery data, mechanical analogues, composed of dashpots and springs, have proved to be useful. The spring is considered as an ideal solid element obeying Hooke's law ($\sigma=G\gamma$) and the dashpot represents an ideal fluid element obeying Newton's law ($\sigma = \mu\dot{\gamma}$). The most common mechanical analogues of rheological behaviour are the Maxwell and Kelvin-Voigt models (Figure 1.12). To model creep curves of viscoelastic materials, the Burgers model which is a combination of a Kelvin-Voigt and a Maxwell model placed in series, is often used (Figure 1.12).

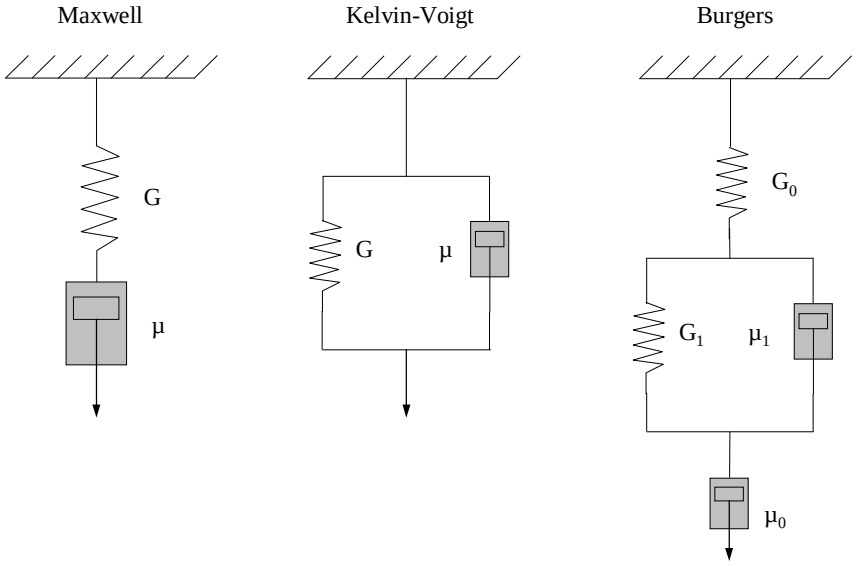

Figure 1.12 Maxwell and Kelvin-Voigt mechanical analogues and their combination in the Burgers model (Based on Steffe, 1996)

Data following the Burgers mechanical analogue (Figure 1.13) show an immediate elastic response due to the free spring, retarded elastic behaviour related to the parallel spring-dashpot combination, and a continuous deformation with a constant shear rate due to the free dashpot (Mezger, 2002; Steffe, 1996).

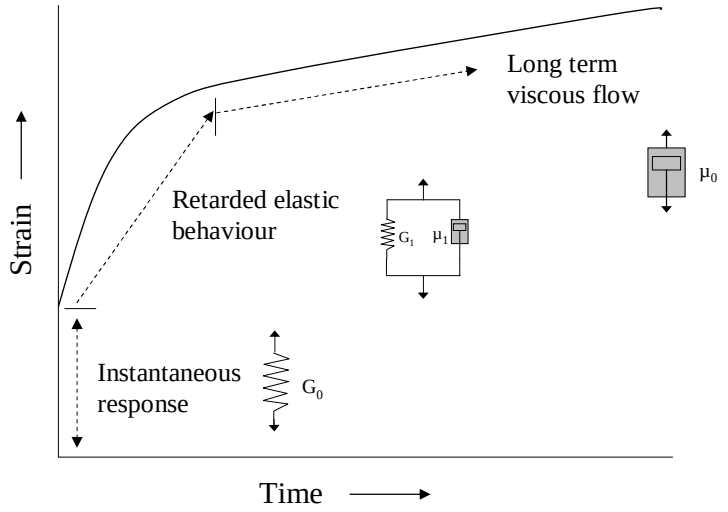

Figure 1.13 Typical creep curve and relation with the various elements of the Burgers model (Steffe, 1996)

The mathematical expression of the Burgers model is given by:

$$\gamma = f(t) = \frac{\sigma_0}{G_0} + \frac{\sigma_0}{G_1}\left(1 - exp\left(\frac{-t}{r}\right)\right) + \frac{\sigma_0 t}{\mu_0} \qquad \textbf{(1-16)}$$

with t: time; γ: total strain; σ_0: constant shear stress; G_0: shear modulus of first spring; G_1: shear modulus of spring or Kelvin-Voigt element; $r = \mu_1/G_1$: retardation time of Kelvin-Voigt element = ratio of the dashpot viscosity and spring shear modulus; μ_0: Newtonian viscosity of the free dashpot.

Ideal elastic materials display an immediate reformation after loading and the subsequent removal of the load. For viscoelastic materials this elastic behaviour occurs with a certain time delay. For tests which use a preset shear stress, this is called retardation which can be described as the delayed response to an applied force or stress or as 'delay of elasticity' (Mezger, 2002). The retardation time (r) is the time constant which determines the time-dependent deformation behaviour of the parallel connected components spring and dashpot in the Kelvin-Voigt model (Mezger, 2002).

The Burgers model can also be expressed in terms of creep compliance by dividing (1-16) by the constant stress:

$$\frac{\gamma}{\sigma_0} = f(t) = \frac{1}{G_0} + \frac{1}{G_1}\left(1 - exp\left(\frac{-t}{r}\right)\right) + \frac{t}{\mu_0} \qquad \textbf{(1-17)}$$

Or writing (1-17) as a creep compliance function:

$$J = f(t) = J_0 + J_1\left(1 - exp\left(\frac{-t}{r}\right)\right) + \frac{t}{\mu_0} \quad \textbf{(1-18)}$$

where J_0 is the instantaneous compliance and J_1 is the retarded compliance.

If necessary, additional Kelvin-Voigt elements can be added to better represent experimental data. Mathematically, this is described by:

$$J = f(t) = J_0 + \sum_{i=1}^{m}\left[J_i\left(1 - exp\left(\frac{-t}{(r)_i}\right)\right)\right] + \frac{t}{\mu_0} \quad \textbf{(1-19)}$$

with m the total number of Kelvin elements in the model, each having a unique retarded compliance and retardation time (Steffe, 1996). In polymer science, each one of these individual Kelvin-Voigt elements stands for the behaviour of one individual polymer fraction with a specific molar mass and a specific molecular structure (Mezger, 2002).

When the applied stress is removed, the sample can recover from the deformation. The recovery can be divided in two parts: an instantaneous elastic recovery related to the free spring and a delayed elastic recovery related to the Kelvin-Voigt elements. The free dashpot in the Burgers model has caused a permanent deformation which can not be restored.

To describe the recovery phase a similar equation as (1-19) can be constructed:

$$J_r = f(t) = J_0 + \sum_{i=1}^{m}\left[J_i\left(1 - exp\left(\frac{-t}{(r)_i}\right)\right)\right] \quad \textbf{(1-20)}$$

Since Bloksma (1962) introduced creep-recovery measurements to study dough rheology, they have been used for determining viscoelastic properties of doughs, batters and gluten. Table 1.4 gives an overview of creep-recovery methodologies used in cereal science research. As can be observed, a wide variety of creep stresses, creep times and recovery times are applied. Creep-recovery measurements are both applied in or outside the LVR. Lefebvre (2006) already suggested that creep-recovery is an interesting tool to measure the non-linear rheological properties of bread dough as during processing dough is subjected to large strains

and shear rates which fall beyond the linear domain. To model creep-recovery data, the Burgers model is often applied as can be seen in Table 1.4.

Table 1.4 Creep-recovery methodology used to study viscoelastic properties of doughs, batters or gluten

System	Type*	Stress (Pa)	Creep (s)	in LVR?**	Recovery (s)	Burgers model***	Ref.
Wheat dough	S	14-500	900	no	100	-	Bloksma (1962)
Wheat dough	S	10-120	250	no	5000	-	Hibberd and Parker (1979)
Wheat dough	T	184-328	3000-4800	-	4200	-	Smith and Tschoegl (1970)
Wheat dough	C	636	240	-	240	-	Wang and Sun (2002)
Wheat dough	C	499	60	-	-	4p	Kawai et al. (2006)
Wheat dough	S	5-10	10000	yes	-	6p	Peressini et al. (2008)
Wheat dough	S	50	600	yes	-	6p	Campos et al. (1997)
Wheat dough	S	50/300	60	yes/no	180	4p	Skendi et al. (2010)
Wheat dough	S	0.8	400	-	800	-	Bauer et al. (2003)
Wheat dough	S	0.3-1000	3h	yes/no	9h/12h	-	Lefebvre (2006); Lefebvre (2009); Rouille et al. (2005)
Wheat dough	S	1-6	3h	yes	12h	-	Lefebvre and Mahmoudi (2007)
Undeveloped dough	S	50	300	yes	-	-	Stojceska et al. (2007)
Durum wheat dough	S	100	300	no	-	-	Edwards et al. (1999)
Durum wheat dough	S	10-50	10000	yes	-	6p	Edwards et al. (2003); Edwards et al. (2001)
Durum wheat dough	S	20	10000	yes	-	-	Edwards et al. (2002)
Wheat sourdough	S	400	300	-	300	-	Clarke et al. (2004)
Biscuit dough	S	10	300	yes	300	-	Pedersen et al. (2004)
Chestnut dough	S	50	60	no	180	4p	Moreira et al. (2010)
Amaranth dough	S	100	300	yes	300	-	Houben et al. (2010)
Maize/flaxseed paste	S	5	120	-	120	4p	Wu et al. (2010)
Rice pasta dough	S	750	120	yes	120	6p	Sozer (2009)
Mochi	S	500	120	-	180	4p-6p	Chuang and Yeh (2006a-b)
Gluten-free dough	S	50	60	no	180	4p	Lazaridou et al. (2007)
Gluten-free dough	S	400	100	-	150	4p	Onyango et al. (2009)
Gluten-free dough	S	50	60	-	180	-	Sivaramakrishnan et al. (2004)
Gluten-free batter	S	80	60	yes	140	4p	Onyango et al. (2010)
Gluten free batter	S	3	100	no	200	-	Nunes et al. (2009)
Wheat gluten	S	50/250	100	yes	1200	6p (recovery)	Tronsmo et al. (2003b); Tronsmo et al. (2003c)
Wheat gluten	S	50	300	-	300	-	Schober et al. (2003)
Wheat gluten	S	40	300	yes	300	-	Pedersen and Jorgensen (2007)
Wheat gluten	S	200	300	-	300	-	Clarke et al. (2004)
Wheat gel	C	50	60	yes	60	-	Sasaki et al. (2008)

* S = shear, C = compression, T = Tension
** '-' = not specified
*** 4p = 4 parameter Burgers model; 6p = 6 parameter Burgers model; '-'= no model used

1.3.3.2. Uniaxial extension

Many food processing operations involve extensional deformation. The molecular orientation caused by extension, versus shear, can produce unique food products with a specific behaviour (Steffe, 1996). Extensional flow is prevalent in dough processing steps like mixing, fermentation (bubble growth) and dough sheeting.

The three basic types of extensional flow are uniaxial, biaxial and planar extension (Figure 1.14). During uniaxial extension the material is stretched in one direction with a corresponding size reduction in the other two directions. In planar extension, a flat sheet of material is stretched in the x_1 direction with a corresponding decrease in thickness (x_2) while the width (x_3) remains unchanged (Steffe, 1996). In the biaxial extension a sample is stretched at equal rates in two perpendicular directions in one plane, as in an expanding spherical balloon. Measurement of biaxial extension properties may be achieved by inflation techniques or lubricated compression (Dobraszczyk and Morgenstern, 2003).

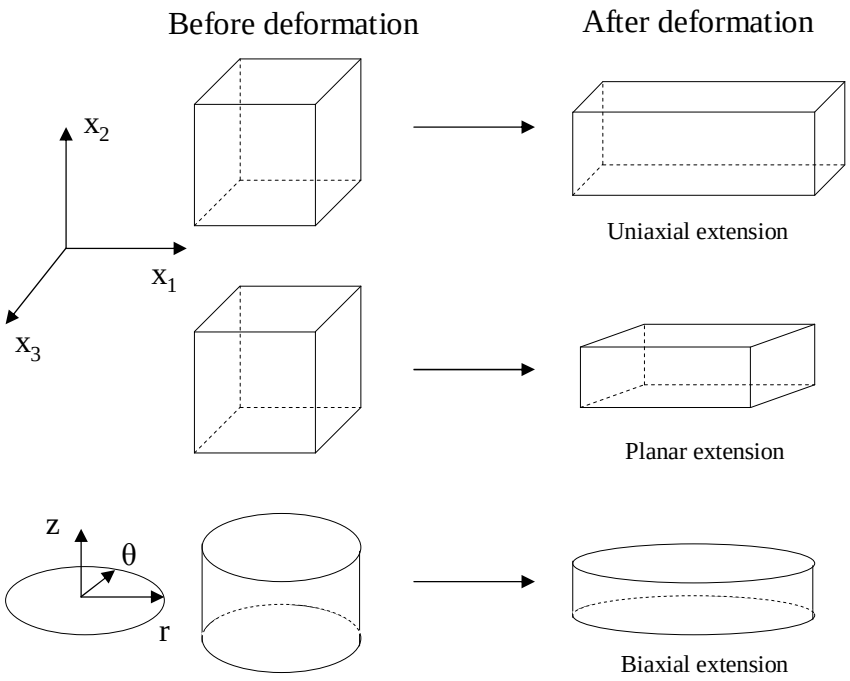

Figure 1.14 Uniaxial, planar and biaxial extension (Steffe, 1996)

Figure 1.15 shows how a material with initial length L_0 is elongated by a tensile force F. The total length after elongation L is the sum of the initial length L_0 and the increase in length ΔL. The deformation can be described as Cauchy strain ε_c:

$$\varepsilon_c = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0} = \frac{L}{L_0} - 1 \qquad \textbf{(1-21)}$$

or Hencky strain (ε_H) which is determined by evaluating an integral from L_0 to L:

$$\varepsilon_h = \int_{L_0}^{L} \frac{dL}{L} = \ln(L / L_0) \qquad \textbf{(1-22)}$$

Cauchy and Hencky strains are both zero when the material is unstrained and approximately equal at small strains. Hencky strain is preferred for calculating strain resulting from a large deformation (Steffe, 1996).

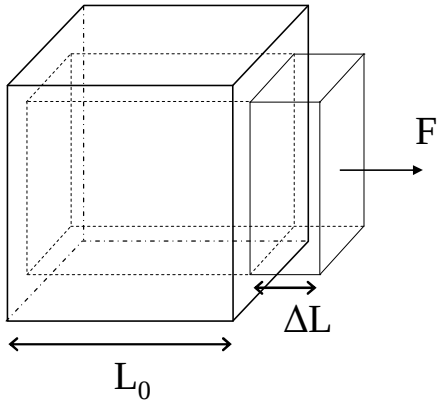

Figure 1.15 Extensional deformation of a material by a tensile force

Consider the uniaxial extension of a material where one end is stationary (glued or clamped) and the other end is moved at a certain velocity v. As the differential Hencky strain describing the displacement is $d\varepsilon_h = dL/L$ (1-22), the strain rate is given by:

$$\dot{\varepsilon}_h = \frac{d\varepsilon_h}{dt} = \frac{1}{L}\frac{dL}{dt} \qquad \textbf{(1-23)}$$

Since dL/dt is the velocity at the end of the sample, the strain rate can be expressed as

$$\dot{\varepsilon}_h = \frac{v}{L} \qquad \textbf{(1-24)}$$

When the velocity is held constant during the test, then the strain rate will continuously decrease during extension. The experimental setup and the mathematical description of the method used for measuring the uniaxial extension properties of bread dough are presented in more detail in section 2.2.4.3.

1.4. Breadmaking process

1.4.1. Introduction

Around 2600 BC, the ancient Egyptians already used sourdough for leavening bread. Although breadmaking is a millennia old process, the principles of breadmaking are still the same today (Dobraszczyk, 2005). The main aim of all breadmaking processes is to transform wheat flour into an aerated, tasty and edible bread product (Cauvain, 2007). Breadmaking processes differ widely in the ingredients, equipment and time/temperature conditions during dough processing and baking, which results in a wide variety of bread products. However, common processing steps for every breadmaking process are mixing, dough shaping, fermentation and baking.

1.4.1.1. Ingredients

The four main ingredients of bread are flour, water, yeast and salt. As already discussed wheat flour and water are essential to obtain a bread dough with the desired viscoelastic properties for gas retention. Other cereal flours may be included in the bread formula depending on the type of bread or they are added to enhance the nutritional quality (Dewettinck et al., 2008).

Yeast is needed to convert fermentable carbohydrates into carbon dioxide and ethanol. The gas that results from this conversion provides the lift that produces a light, leavened loaf of bread (Delcour and Hoseney, 2010). In some countries, a sourdough is commonly applied as leavening agent.

Salt has an important role in breadmaking which includes stabilizing yeast fermentation rate, strengthening the dough, increasing mixing time and enhancing flavour of the final product.

However, preparing a high, consistent bread quality from only these four raw materials is difficult. For this reason several functional additives are added for improving dough properties and bread quality. A wide range of additives is available e.g. vital gluten, emulsifiers, enzymes, oxidising and reducing agents and hydrocolloids which all have a

specific functionality in breadmaking (De Leyn, 2006; Goesaert et al., 2005; Joye et al., 2009). Other ingredients commonly used in breadmaking are oils and fats, sugar and milk products.

1.4.1.2. Breadmaking systems

According to the way of dough development, three major breadmaking systems can be recognized: the straight dough system, the sponge and dough system, and short time breadmaking systems (Cauvain, 2007; Delcour and Hoseney, 2010). The three breadmaking systems are shortly discussed below, a more extended overview has been provided by Cauvain (2007).

In the straight dough system, ingredients are mixed to form a homogeneous dough where after the dough is bulk fermented for a prescribed time. During the bulk fermentation period there may be a remixing of the dough, also called punching. After the bulk fermentation period, the dough is divided, moulded and placed into a pan. After an additional fermentation period (proof), the dough is baked. The straight dough process is commonly used in Europe but is known to yield bread with a coarser cell structure and less flavour.

The sponge and dough process is a two-step process in which first a part of the total quantity of flour, water and other ingredients is allowed to ferment (the sponge). The remainder of the ingredients and the sponge are then mixed into a homogeneous dough and after a short period of bulk fermentation, the dough is further processed as in the straight dough system. This process is the most popular in North-America.

Short time breadmaking systems were developed to reduce the time needed for breadmaking. In processes such as activated dough development extra ingredients are added to assist dough development and to reduce the fermentation periods. In mechanical dough development, the dough is fully developed in the mixer, thus making bulk fermentation unnecessary. The Chorleywood Bread Process (CBP) is the best known breadmaking process based on mechanical dough development and is the most used process in the UK.

1.4.2. Breadmaking steps

1.4.2.1. Mixing

Mixing is a crucial stage in all breadmaking processes. In the mixing process, wheat flour components and other ingredients are hydrated and blended into a homogeneous mass (Ktenioudaki et al., 2010a). In the early stages of mixing, flour particles become hydrated and gradually cohere together into an undeveloped dough. Upon further mixing, a rise in consistency is observed which indicates the development of the viscoelastic gluten network (Stauffer, 2007). Further, mixing achieves the aeration of the dough, which provides the bubbles which evolve into final bread crumb cells (Martin et al., 2004).

Dough mixers can be found in several types and sizes. Mixers differ in their mixing action and the intensity of the mixing action, i.e. the amount of energy worked into the dough. Mixers are usually divided into low, medium and high intensity mixers (Dobraszczyk, 2005; Marsh and Cauvain, 2007). The extent of mixing or the amount of energy required to develop a dough depends on various factors, including mixing speed, mixer design and flour characteristics (Chin and Campbell, 2005a). According to Kilborn and Tipples (1972), there are two basic requirements to achieve proper development of a dough: mixing intensity (impeller speed) must be above a minimum critical level that varies with both flour and mixer; and the work imparted to the dough must be greater than a minimum critical amount dependent on the flour used.

For each combination of flour and mixer it is possible to find an optimum stage of dough development (Eliasson and Larsson, 1993). Best baking results are obtained with doughs that have been mixed just past the maximum of the consistency curve (Wrigley et al., 2006). Recording mixers such as the mixograph or the farinograph are widely used to analyze dough mixing properties (Haraszi et al., 2008).

1.4.2.2. Fermentation

During fermentation, the yeast cells will convert fermentable sugars into carbon dioxide, ethanol and energy. The carbon dioxide dissolves in the dough and eventually diffuses to the gas bubbles. A gradual increase in pressure of the gas cells will cause expansion of the dough. Next to gas production, yeast fermentation has a pronounced effect on dough rheology and the flavour of the bread (Cauvain and Young, 2007).

1.4.2.3. Dough processing

The next stages in bread manufacture are the subdivision of the bulk dough (dividing) and the shaping of individual dough pieces (moulding) to obtain the desired bread variety. Shaping is a multi-stage operation and may involve a further resting period between moulding stages (intermediate or first proof) (Marsh and Cauvain, 2007). Dough processing is further discussed in Chapter 6.

1.4.2.4. Baking

During baking, a series of chemical and physical changes occur which transform the dough into bread. When a dough is placed in the oven, three large changes are seen: an immediate expansion of the dough (oven rise), drying of the surface and eventually crust formation and browning. Oven spring is the result of extra gas production by yeast before a temperature of 55°C is reached, expansion of the gas in the dough due to the rise in temperature, lower solubility of carbon dioxide in the dough and vaporization of the water-ethanol azeotrope. The heat transferred to the dough causes starch gelatinization and protein denaturation which results in the formation of the bread crumb (Cauvain and Young, 2007; Delcour and Hoseney, 2010).

1.5. Dough rheology and baking potential

Rheological properties of dough are important to the baker for two reasons. First of all, they determine the behaviour of dough pieces during mechanical handling, such as dividing, rounding and moulding. Secondly, they affect the quality of the finished loaf of bread (Bloksma and Bushuk 1988).

MacRitchie (2003) pointed out that the expansion capacity of a dough is determined by balanced dough rheological properties and gas cell stability. Bloksma (1990b) stated that a dough for producing high quality loaves, should meet two requirements: (1) the dough must have a sufficiently high viscosity to prevent the ascent of gas cells and (2) it must remain extensible long enough during baking to avoid premature rupture of membranes between gas cells. Extensibility has indeed been related to baking performance (Bettge et al., 1989;

Ktenioudaki et al., 2010b; Suchy et al., 2000) but others showed that the resistance against extension was more important (Dobraszczyk and Salmanowicz, 2008; Kieffer et al., 1998). However, the area under the extension curve, indication of the ratio of strength and extensibility, may be a better predictor for bread quality (Dowell et al., 2008; Nash et al., 2006). It can be concluded that to ensure stability of gas cells, the dough needs to be sufficiently extensible to respond to gas pressure but also strong enough to resist collapse (Sroan et al., 2009).

Several researchers have tried to find a link between dough rheology and baking performance, or more specifically, bread volume. Table 1.5 gives an overview of correlations found between fundamental rheological properties and baking volume.

Table 1.5 Fundamental rheological properties related to bread volume

	Product	*#samples*	*Parameter*	*Correlation*[*]	*Ref.*
Dynamic oscillation	dough	24	G'	r=0.15	Autio et al. (2001)
			G' (all points stress sweep)	r=0.28	
			delta	r=0.72	
	dough	12	G'	n.s.	Khatkar and Schofield (2002)
	spelt gluten	11//25	$\|G^*\|$	r= -0.855// -0.568	Schober et al. (2002)
	gluten	40	moduli/delta	n.s.	Tronsmo et al. (2003c)
	gluten	12	G'	r^2=0.73	Khatkar and Schofield (2002)
Creep-recovery	gluten	40	%recovery	r=0.62	Tronsmo et al. (2003c)
	dough	24	creep strain	r=0.1	Wang and Sun (2002)
			recovery strain	r>0.9	
Uniaxial elongation	dough (varying protein content	22	rupture strain	r=0.66	Uthayakumaran et al. (2000)
			rupture viscosity	r=-0.59	
	dough (varying glu/gli ratio)	10	rupture strain	n.s.	Uthayakumaran et al. (2000)
			rupture viscosity		
Biaxial extension	dough	6	strain hardening	r=0.92-0.97	Dobraszczyk et al. (2003)
			failure strain		
	dough	36	strain hardening	r=0.88	Dobraszczyk and Salmanowicz (2008)
			failure strain	r=0.89	
	dough	9	strain hardening	r=0.7	Ktenioudaki et al. (2010b)

[*] n.s. = non significant

Small deformation oscillation experiments are widely used to study dough rheology. However, no convincing relationship with baking performance has been established for wheat flour dough. Better results were obtained for isolated gluten (Khatkar and Schofield, 2002; Schober et al., 2002). It is believed that dynamic oscillation tests are not suited to elucidate baking performance as the small deformations used in these tests are not relevant for the deformations occurring during fermentation and dough processing (Dobraszczyk and Morgenstern, 2003). Although no direct relationship has been found with bread volume, correlations have been reported between the form ratio of hearth bread and dynamic oscillation and creep-recovery parameters of gluten (Schober et al., 2002; Tronsmo et al., 2003b).

A more recent concept for explaining breadmaking performance of a wheat flour is strain hardening. A review on the topic was recently presented by van Vliet (2008). A bread dough shows strain hardening when subjected to large deformations such as bubble expansion or compression. Strain hardening is the non-linear increase of stress with increasing strain giving rise to a typical J-shaped stress–strain curve (Dobraszczyk and Morgenstern, 2003). At a molecular level, strain hardening can be explained by the well established polymer entanglement network theory. It is believed to originate from entanglement coupling of large glutenin chains (Singh and MacRitchie, 2001). Strain hardening allows the expanding gas cell walls to resist failure by locally increasing resistance to extension as the bubble walls become thinner, and appears to provide the bubbles with greater stability against early coalescence and better gas retention. Several authors have reported that strain hardening was important in baking performance (Dobraszczyk and Roberts, 1994; Dobraszczyk et al., 2003; Janssen et al., 1996b; Kokelaar et al., 1996; Sliwinski et al., 2004b; van Vliet et al., 1992). It has been reported that breadmaking varieties of good quality show greater strain hardening and extensional viscosity (Dobraszczyk and Morgenstern, 2003; Dobraszczyk and Salmanowicz, 2008; Sroan et al., 2009). However, strain hardening could not explain the differences in baking volume caused by varying flour lipid quantity and composition (Sroan and MacRitchie, 2009).

Instead of using only one parameter to predict baking performance of wheat flour, several authors have succeeded to obtain better correlations with bread volume by combining chemical and rheological parameters through regression analysis (Dobraszczyk and

Salmanowicz, 2008; Dobraszczyk et al., 2005; Dowell et al., 2008; Kieffer et al., 1998; Ktenioudaki et al., 2010b; Wikstrom and Bohlin, 1999).

Based on a dataset of 36 wheat samples, Dobraszczyk and Salmanowicz (2008) reported that the best subset of parameters to predict baking volume using dough inflation was a combination of strain hardening index, bubble failure strain and protein content (r^2=0.865). From the Kieffer extensibility parameters, the best subset consisted of maximum resistance, area under the extensibility curve and protein content (r^2=0.842). No subsets were reported combining parameters obtained from different rheological test methods. Ktenioudaki et al. (2010b), on the other hand, found the best prediction of the bread volume by combining maximum uniaxial extensibility and biaxial extensional viscosity (r^2=0.777). Wikstrom and Bohlin (1999) combined 6 parameters obtained from uniaxial extensional flow measurements with protein content and Zeleny sedimentation value and could so explain up to 97% of the variation in bread volume. However, it was not indicated if all variables contributed significantly to the regression model. Protein content and wet gluten content were also used in combination with Kieffer extensibility parameters to improve the correlation with bread volume (Kieffer et al., 1998). In the study of Dowell et al. (2008), the baking volume of 48 hard red winter and 49 hard red spring varieties was largely determined by protein content (r^2=0.85). The prediction could be improved further (to r^2=0.91) by including parameters obtained from chemical composition (%gliadins or %glutenins), farinograph analysis and kernel properties. No fundamental rheological properties were determined in this study.
Thus, combining the protein content of the wheat flour with a rheological parameter of the dough system, generally results in the best prediction models for bread volume.

Chapter 2

Materials and Methods

MOLJER: maalder.

KETMEEL: kortmeel, pellen van gemalen graan.

[Oilsjtersen Diksjoneir]

2.1. Materials

The wheat flours used in this thesis are listed in Table 2.1. The quality description will be given in the respective chapter. All flour samples were stored at -20°C.

Table 2.1 Wheat flour types and origin with indication of corresponding chapter

Chapter	*Name*	*Producer*
3	Bussard (1)	Paniflower
4	Bussard (1)	Paniflower
	Wheat cultivars (see Table 4.1)	University College Ghent
5	Epi B	Paniflower
6	Bussard (2)	Paniflower
	Tulsa	Clovis Matton/ University College Ghent

Flours produced at University College Ghent were milled on a Bühler laboratory mill. Before milling, wheat samples were cleaned and tempered overnight to a moisture content of 15.5%.

2.2. Methods

2.2.1. Chemical analysis

Moisture content, protein content and ash content were determined according to ICC official methods 110/1, 105/2 and 104/1 respectively (ICC, 2005).

2.2.2. Flour quality parameters

Hagberg Falling Number, Zeleny sedimentation value and wet gluten content were determined according to ICC official methods 107/1, 116/1 and 137/1 respectively (ICC, 2005). Whereas damaged starch content was determined according to AACC official method 76-31 (AACC, 1990).

2.2.3. Determination of the high molecular weight glutenin subunit (HMW-GS) composition of wheat flour

For determining the composition of high molecular weight glutenin subunits (HMW-GS), the Invitrogen XCell Surelock Mini-Cell electrophoresis apparatus was used with two pre-cast gel types, a NuPAGE 4-12% bis-tris gel (10 x 10 x 0.15 cm) and a 10% tris-glycine gel (10 x 10 x 0.10 cm). These two types of gels were applied to be able to separate all HMW-GS as this can not always be achieved by only using the common tris-glycine system (Kasarda et al., 1998).

All reagents and pre-cast gels were purchased from Invitrogen (Merelbeke, Belgium). For both gel types, samples were prepared following the manufacturer's protocol. For the NuPAGE 4-12% bis-tris gels, 10 mg of flour was added to 250 µL NuPAGE LDS sample buffer, 100 µL NuPAGE reducing agent and 650 µL deionized water. The samples were vortexed and heated to 70°C for 10 min in a hot water bath. Then 10 µL of each sample was loaded onto the gel and a 3-(N-morpholino) propane sulfonic acid buffer (MOPS, 50 mM, pH 7.7) was used as running buffer. To the load buffer, 500 µL NuPAGE antioxidant was added. Constant voltage separation was performed in 70 min at 150 V. In the case of the 10% tris-glycine gels, 10 mg of flour was added to 500 µL tris-glycine SDS sample buffer, 100 µL NuPAGE reducing agent and 400 µL deionized water. The samples were vortexed and heated to 85°C for 2 min in a hot water bath. Then 8 µL of each sample was loaded onto the gel and tris-glycine SDS running buffer was used. Constant voltage separation was performed in 30 min at 150 V and 75 min at 200 V. After separation, gels were washed three times with deionized water and stained for 60 min with Coomassie Blue stain. The gel was destained in deionized water for 60 min. Scanning of the wet gel was done with a high-resolution transmission scanner (UMAX Powerlook III). Gels were analyzed with the Imagemaster Totallab software (Amersham Biosciences, Roosendaal, The Netherlands).

The HMW-GS were numbered according to Payne and Lawrence (1983) and a score for Glu-1 quality was calculated (Payne et al., 1987). To enhance the assignment of the HMW-GS, reference wheat flours with a known HMW-GS composition were analyzed together with the unknown samples. Wheat flours with known HMW-GS composition were Bussard (1, 7+9, 5+10), Albatros (null, 7+8, 2+12), Moulin (null, 17+18, 2+12) and Granada (2*, 6+8, 5+10). An example of the SDS-PAGE for two wheat cultivars with different composition is shown in Figure 2.1.

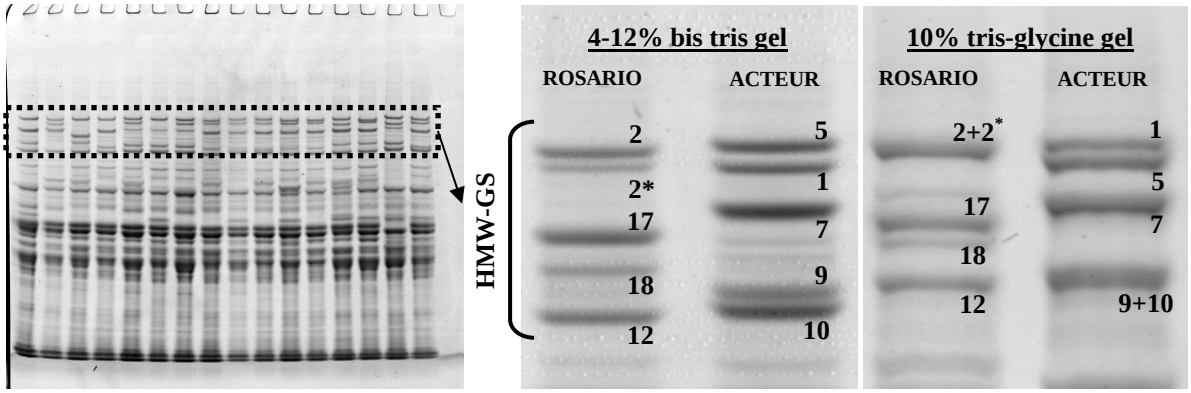

Figure 2.1 SDS-PAGE to identify the HMG-GS composition: a complete gel and a detail of the HMW-GS composition of two wheat cultivars (Rosario and Acteur) from two gel types

2.2.4. Dough rheology

2.2.4.1. Farinograph

The farinograph is essentially a torque-measuring, recording dough mixer. It mixes flour and water into a dough, develops it and finally overmixes the dough (D'Appolonia and Kunerth, 1984). Dough is developed in the farinograph mixer bowl (Figure 2.2) by two z-shaped mixing arms which rotate at different speeds (63 and 92 rpm) towards each other. Different mixer bowl sizes are available (50 and 300 g). Dough resistance to mixing is recorded and a farinogram is obtained (Figure 2.2).

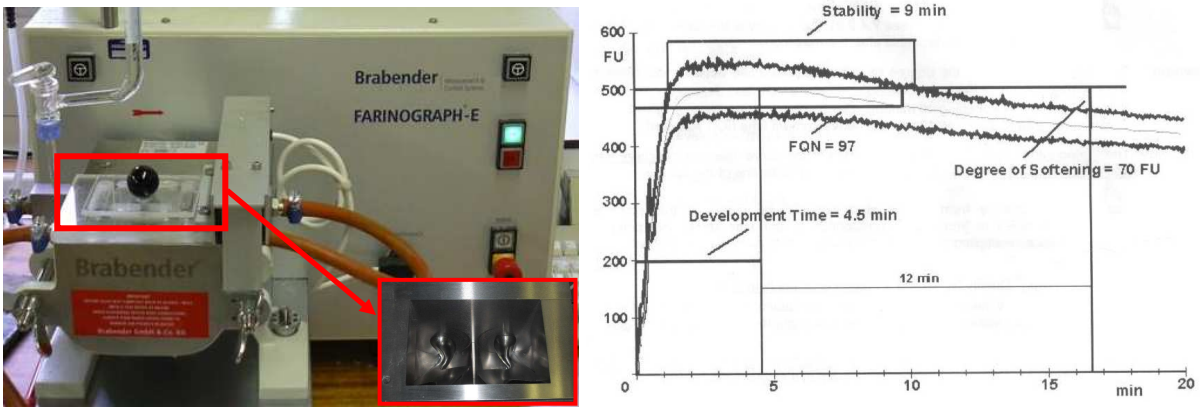

Figure 2.2 Farinograph recording mixer and an example of a farinogram (adapted from Brabender farinograph operating manual)

The method for performing a farinograph test on wheat flour is well described in ICC or AACC standard methods (Table 1.2). Parameters obtained from the farinograph mixing curve

are water absorption (WA) to reach a dough consistency of 500 FU (farinograph units) and corrected to a flour moisture content of 14%, dough development time (DDT), dough stability (STAB), dough softening (SOFT), elasticity (ELAST) and farinograph quality number (QUAL). The elasticity is the bandwidth at the curve maximum. All reported values in this research are the mean value of at least three replicates.

2.2.4.2. Alveograph

The Chopin Alveograph is a dough testing instrument which was developed in the 1920s and 1930s to replace baking tests for assessing the baking quality of French wheats (Dubois et al., 2008). In the standard alveograph test, a dough is mixed with a fixed amount of salt water (50%) and extruded to obtain separate dough pieces. The dough pieces are then inflated into a bubble until rupture to evaluate dough rheological properties (Figure 2.3). The alveograph test procedure is well described in ICC or AACC standard methods (Table 1.2).

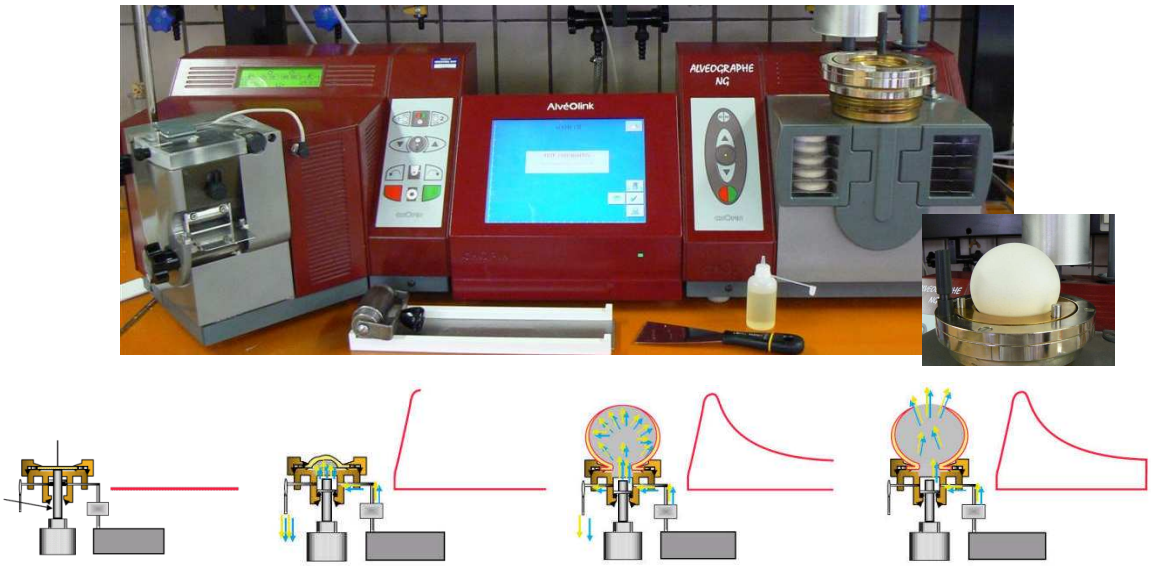

Figure 2.3 The alveograph NG instrument and working principle of dough inflation until rupture with registration of the alveogram (inflation graphs adapted for Chopin Technologies)

Parameters obtained from the alveogram are tenacity or resistance to extension (P), dough extensibility (L), curve configuration ratio (P/L) and deformation energy (W) which corresponds to the area under the curve. The elasticity index (I_e) is given by P_{200}/P in which P_{200} is measured when L equals 40 mm. All reported values in this research are the mean value of at least three replicates of which five dough pieces were inflated.

2.2.4.3. Kieffer dough and extensibility rig

The 'Kieffer dough and gluten extensibility rig', also called the micro-extensograph, was developed as an alternative to the Brabender extensograph (Kieffer et al., 1981). The Kieffer rig consists of three main parts: the sample preparation press and mould, the spring-loaded test rig and the hook with PTFE sleeve. First, a dough is pressed in the mould to provide a series of small dough strips. After a relaxation period, the dough strips are removed from the mould and clamped at both ends onto the test platform. The dough piece is extended upwards until breakage occurs. The force needed to extend the dough piece is recorded as function of the stretching length or time (Figure 2.4).

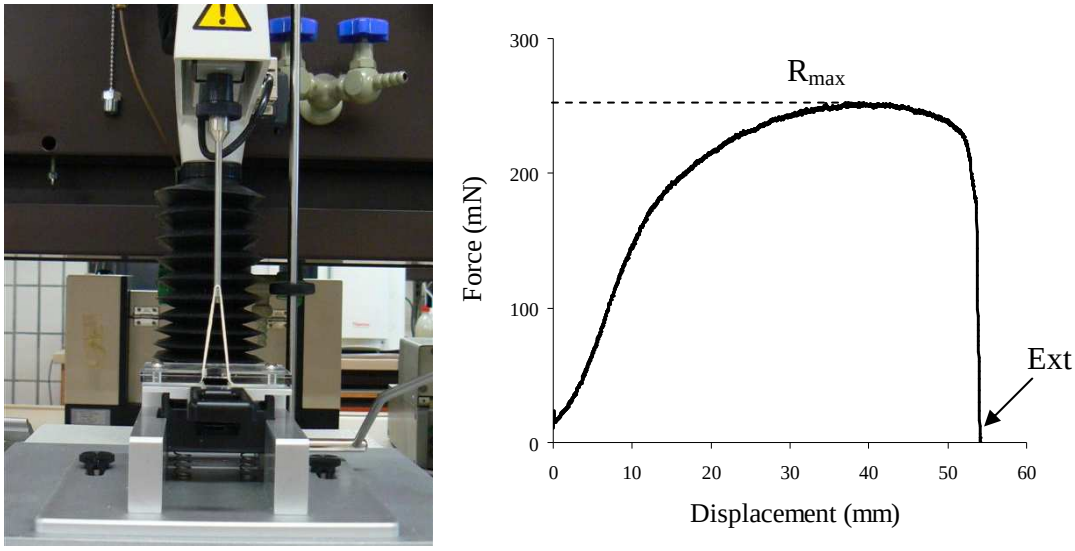

Figure 2.4 Experimental setup of the Kieffer dough and gluten extensibility rig on the TA.XTplus texture analyzer and an example of a force-displacement curve with indication of maximum extensibility (Ext) and maximum resistance (R_{max})

As no standard method is available, the test procedure was based on the methods described by Smewing (1995) and Peighambardoust et al. (2006). Measurements were made on a TA.XTplus texture analyzer equipped with a Kieffer dough and gluten extensibility rig which was placed in a thermostatic cabinet at 26°C. Freshly prepared dough samples which rested for 10 min at 26°C and 90% relative humidity, were placed on the grooved Kieffer mould and compressed to obtain thin dough strips. The mould was placed at 26°C and 90% RH for 45 min which allowed the dough to relax prior to measurement. Dough strips were extended with a hook speed of 3.3 mm/s until fracture. For each dough, five strips were analyzed. At least three independent replicates, each from a separately mixed dough, were performed.

Parameters obtained from the force-displacement curves are the maximum resistance to extension (R_{max}), maximum extensibility (Ext) and the area under the curve (Area).

Fundamental rheological parameters can be obtained out of the force-displacement curves from uniaxial extension measurements performed with the Kieffer dough and gluten extensibility rig. The theoretical background has been described by Dunnewind et al. (2004). A schematic drawing of the Kieffer extensibility rig and the forces acting on the dough piece are shown in Figure 2.5.

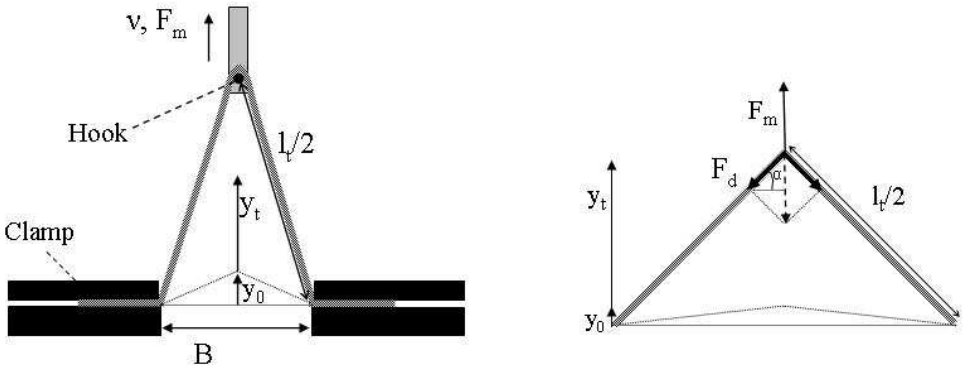

Figure 2.5 Schematic drawing of the Kieffer extensibility rig and the forces acting on the dough piece (adapted from Dunnewind et al., 2004)

When the sample is clamped between the two plates, a small amount of dough is squeezed out of the plates and the sample shows some sagging due to gravity. This means that the point where actual extension starts, is situated somewhere above the surface of the lower plate. The distance to reach this point is equal to y_0. The initial length of the sample l_0 and the length l_t at time t are:

$$l_0 = 2.\sqrt{(B/2)^2 + y_0{}^2} \quad \textbf{(2-1)}$$

$$l_t = 2.\sqrt{(B/2)^2 + (y_0 + y_t)^2} \quad \textbf{(2-2)}$$

in which y_t is the displacement of the hook from the point at which extension starts and B is the width of the gap between the clamps.

The Hencky strain and the strain rate can be written as:

$$\varepsilon_H = \ln\left(\frac{l_t}{l_0}\right) = \ln\left(\frac{\sqrt{(B/2)^2 + (y_0 + y_t)^2}}{\sqrt{(B/2)^2 + {y_0}^2}}\right) \quad \textbf{(2-3)}$$

$$\dot{\varepsilon} = \frac{d\varepsilon_H}{dt} = \frac{dl}{l_t\, dt} = \frac{1}{l_t} \cdot \frac{2(y_0 + y_t)}{\sqrt{(B/2)^2 + (y_0 + y_t)^2}} \cdot \frac{dy_t}{dt} = \frac{4.(y_t + y_0).v}{{l_t}^2} \quad \textbf{(2-4)}$$

The measured force F_m is not the force acting on the dough F_d. Assuming that the hook passes exactly through the centre of the gap, F_m is divided equally over both stretches of the dough at each side of the hook. Sinα can therefore be expressed in forces as well as in lengths:

$$\sin\alpha = \frac{F_m/2}{F_d} = \frac{y_t + y_0}{l_t/2} \quad \textbf{(2-5)}$$

Assuming that the dough piece has the same cross-section over its whole length, the surface over which the force is acting is V/l_t, V being the volume of the dough piece that is extended. The stress σ can be calculated according to:

$$\sigma = \frac{F_d}{V/l_t} \quad \textbf{(2-6)}$$

The force-displacement curves were recalculated into stress-strain data from which the uniaxial extension fracture properties were derived (Figure 2.6): the maximum stress or fracture stress (σ_{max}) and the Hencky strain at fracture stress ε_H. From the stress-strain curves also the strain hardening index (SHI) was calculated by fitting an exponential curve to the stress-strain data (Dobraszczyk and Salmanowicz, 2008; Tronsmo et al., 2003b). For this calculation, the strain interval 20-95% of fracture strain was used and thus an apparent strain hardening value was obtained (Peighambardoust et al., 2006).

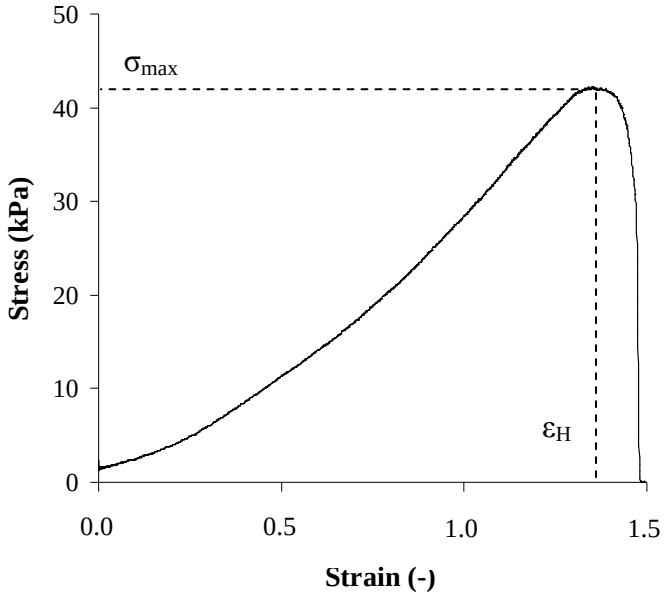

Figure 2.6 Example of a stress-strain curve for an extension experiment

2.2.4.4. Rotational rheometry

The methodology for using the rheometer to analyze dough rheology is described in Chapter 3.

2.2.5. Breadmaking tests

2.2.5.1. Large-scale breadmaking test

The large scale breadmaking test for evaluating the baking performance of wheat flour is a straight dough baking method, based on the ICC standard method 131 (Vanneste and De Leyn, 2004). A flow chart of the breadmaking procedure is presented in Figure 2.7.

The bread formula contained wheat flour (2 kg, 14% moisture), dry instant yeast (1%), salt (1.5%), ascorbic acid (25 ppm), tap water and malt flour. The amount of malt flour was adapted in order to adjust the falling number of the flour to 250. The amount of water was based on the farinograph water absorption of which only 95% was added to the flour to improve the processability of the dough. Salt and ascorbic acid were dissolved in the water, while malt flour and yeast were blended with the flour. The dough was prepared by mixing in

a spiral mixer (type De Danieli IS.06) for 7 min. The temperature of the dough after mixing was 26.5-27.5 °C by adjusting the temperature of the added water.

After mixing, the dough was placed in the fermentation cabinet (30 °C – 85% rh) for 10 min. Following this resting period, the dough was divided into six pieces of 400 g, the dough pieces were rounded with a Brabender rounder and placed in the fermentation cabinet for 30 min. Following the first fermentation period, the dough pieces were moulded, placed into baking pans and fermented for another 65 min.

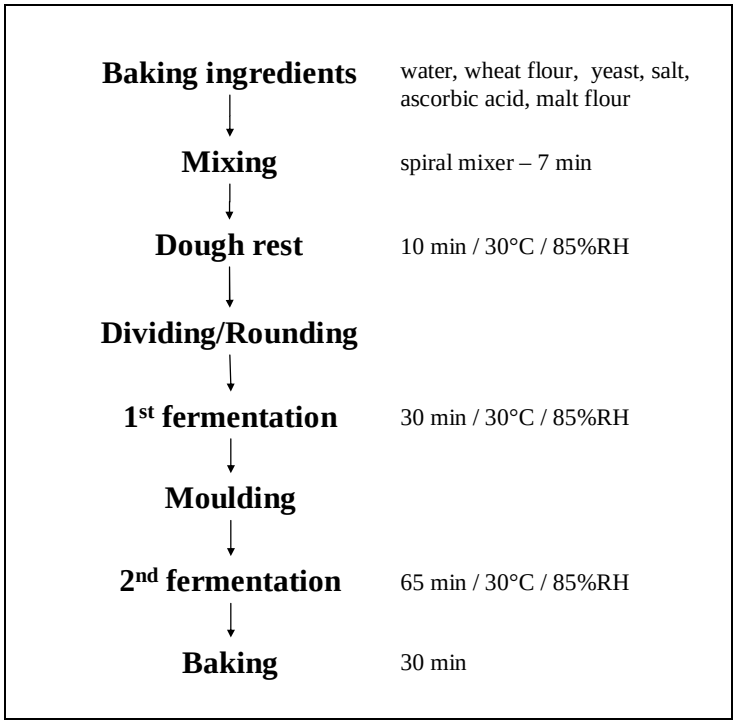

Figure 2.7 Flow chart of the large scale breadmaking procedure

The loaves were baked in the oven for 30 min with steam injection at the start of the baking program (240 mL water in 2 min). The temperature program during baking consisted of 3 consecutive phases: 14 min at 230 °C, 13 min at 200 °C and 3 min at 190 °C during which the steam valve was opened. Breads were cooled down to room temperature before the volume was measured by the rapeseed displacement method. The reported baking volumes (recalculated to 100 g flour) are the average of at least 5 bread volumes corresponding with one baking test. The breadmaking equipment is shown in Figure 2.8.

Figure 2.8 Breadmaking equipment: A. spiral mixer, B. Brabender rounder, C. Oven, D. Fermentation cabinet

2.2.5.2. Small-scale breadmaking test

A small scale breadmaking procedure was developed based on the standard breadmaking procedure as described in 2.2.5.1. Adaptations were made concerning the mixer type, weight of the dough pieces and the baking time.

For the small scale breadmaking test, doughs were mixed in a small scale mixer like the 300 g farinograph mixing bowl. The mixing time is not fixed and can be chosen according to the desired dough development. The advantage of using the farinograph mixing bowl, is that dough development during mixing can be recorded.
From the mixed dough, two pieces of 125 g and two pieces of 100 g dough were obtained. Those four dough pieces were processed identically as described in 2.2.5.1. For the final fermentation period, the pieces of 125 g are placed in baking pans and the pieces of 100 g are placed on U-profile plates to simulate hearth bread.
As dough pieces were smaller, the baking program was shortened to 20 min. The temperature program during baking consisted of 3 consecutive phases: 2 min at 230 °C, 13 min at 200 °C and 5 min at 230 °C during which the steam valve was opened.

After baking, the breads were cooled down to room temperature before weighing and volume determination with the rapeseed displacement method. For the plate bread, the height over width ratio (H/W) was calculated. This ratio gives information about the flow of the dough during fermentation and oven rise. The reported results are the average of three baking tests in which two breads of both types (pan and plate bread) were obtained.

For calculating oven rise, dough height before baking and the height of the bread were measured.

2.2.6. Visualisation of dough microstructure

Confocal scanning laser microscopy (CSLM) is a powerful tool for the visualisation of the structure of biopolymer mixtures and food products. The key feature of CSLM is the imaging of a single focal plane in a sample at a depth in the micrometer range. The fluorescent light from the fluorescent dyes in the sample is collected by the objective lens and focused into a small pinhole to eliminate the out-of-focus light. Because of this pinhole, the confocal microscope provides excellent resolution within the focal plane. An optical section of the sample is obtained by a point by point scanning of the sample in the x- and y-direction within the focal plane (van de Velde et al., 2003).

To visualize starch and proteins in the dough structure simultaneously, a double staining technique was used as described by Peighambardoust et al. (2004, 2006). A mixture of fluorescein isothiocyanate (FITC) and Rhodamine B (1 and 0.1% w/v, respectively) in dimethylformamide was used for non-covalent labelling of starch and proteins, respectively. The stained doughs were stored at ambient temperatures for 1-2 h before observation.

Visualisation of the dough microstructure was carried out with a Nikon Eclipse TE300 (Analis, Belgium) epifluorescence microscope connected to a Biorad Radiance 2000 (Bio-rad Laboratories, Belgium) confocal system. A 10x microscopic objective was mostly used. FITC was excited by a 488 nm argon-ion laser and its fluorescence detected through a 515/30 filter. Rhodamine B was excited by a 543 nm green helium-neon laser and detected through a 600LP filter. The laser intensity, gain, and offset were chosen in order to prevent oversaturation of the fluorescence signal. Kalman filtering was used to diminish the background noise on the picture. Imaging was performed in an air-conditioned room (18-20°C). An overlay of the obtained images was performed by using ImageJ freeware. An example of a CSLM image for bread dough is shown in Figure 2.9.

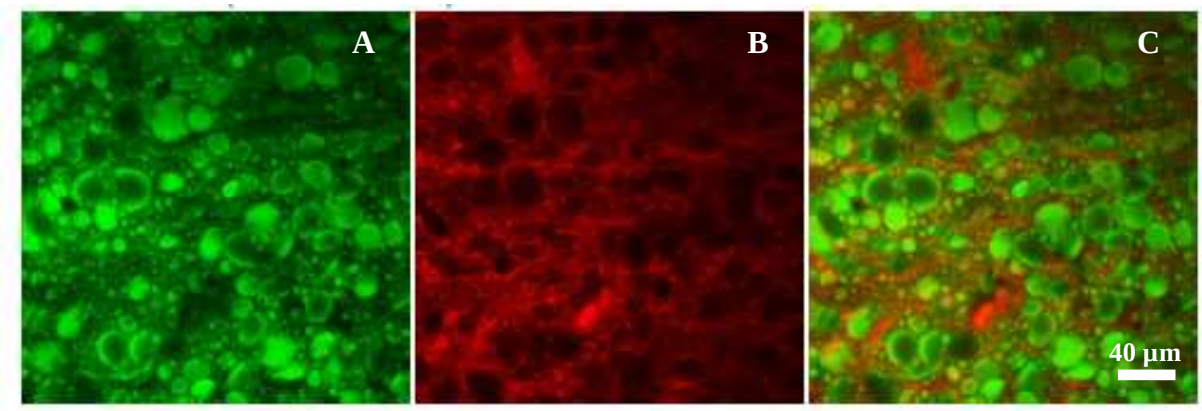

Figure 2.9 CSLM images of bread dough obtained from excitation of FITC at 488nm (A), excitation of rhodamine at 543 nm (B) and the merged image (C)

2.2.7. Mathematical and statistical analysis

2.2.7.1. Burgers model

All modelling was performed with the regression wizard of SigmaPlot2000 (SPSS Inc.)

To decide whether to use the conventional four parameter Burgers model or the more complex 6 parameter Burgers model (based on equation 1-19) to model the creep data, a statistical F-test was used. The statistical F-test is probably the most frequently applied method to decide whether the more complex model *j* is significantly (with a significance level α) better than model *i*. The test statistic is calculated as:

$$F_w = \frac{\left(SSR_i - SSR_j\right)/(n_{p_j} - n_{p_i})}{SSR_j/(N_d - n_{p_j})} \quad \textbf{(2-7)}$$

with *SSR* the sum of the squared residuals, n_p the number of parameters in the respective model and N_d the number of data points. The obtained value of the test statistic has to be compared with tabulated values for $F_\alpha(n_{pj}\text{-}n_{pi}, N_d\text{-}n_{pj})$ (Foubert, 2003). α was set to 0.05.

2.2.7.2. Anova

Significant differences were determined with SPSS (SPSS Inc) by one-way Anova (α=0.05). The Shapiro-Wilk test was used to check whether the data were normal distributed. When significant differences were detected, a post-hoc test was executed to evaluate which

treatment groups were significantly different. In case of equal variances, Tuckey was used. When variances were significantly different, Dunnett T3 was used as post-hoc test. The Levene test was applied for evaluating the homogeneity of variances.

2.2.7.3. Pearson correlations

To explore relationships between variables, the Pearson correlation coefficients (r) significant at $P<0.05$ and $P<0.01$ (indicated * and **, respectively) were calculated. Pearson correlations were determined with SPSS (SPSS Inc.).

2.2.7.4. Multiple linear regression

To investigate the effect of different variables on the bread volume in Chapter 4, multiple linear regression with a forward model selection was used. In the forward method, independent variables are entered step by step based on an *F* test. This *F* test is used to decide whether a more complex model is significantly better than the more simple model. In forward model selection the independent variable with the highest *F* value (lowest significance) and thus the highest correlation with the dependent variable is added to the model first. In a second step, the variable which has then the highest *F* value, is added (Foubert et al., 2004). The model is complete when all the variables with a significance value below a maximum value are added. In this research a maximum significance value of 0.05 was used. Forward linear regression was performed with SPSS (SPSS Inc.).

2.2.7.5. Principal component analysis (Foubert, 2003)

Principal component analysis (PCA) is a mathematical procedure that transforms a number of (possibly) correlated variables into a number of uncorrelated variables called principal components. These principal components are linear combinations of the original variables. The coefficients of the original variables in these linear combinations are chosen so that the first principal component accounts for as much of the variability in the data as possible and each succeeding component accounts for as much of the remaining variability as possible. Instead of working with all original variables, PCA can be performed and only the first two or three principal components can be used in subsequent analyses. The objective of PCA is thus

to reduce the dimensionality (number of variables) of the data set while retaining most of the original variability in the data.

PCA was mainly used in this research in Chapter 4 to visualize variability in the dataset of wheat cultivars based on selected quality or rheological parameters. PCA was performed on the standardized variables and principal components with an eigenvalue above 1 were retained. A Varimax rotation was applied to the principal components with an eigenvalue above 1. The aim of this Varimax rotation is to relate each original variable as much as possible to one principal component and as such facilitate the interpretation of the principal components. PCA was performed with SPSS (SPSS Inc.)

2.2.7.6. Standard deviation and adapted t-test (Foubert et al., 2003)

Because the reported values $\overline{\beta}_{(j)}$ of some rheological parameters are the mean value of values which are estimations themselves $\hat{\beta}_{ji}$, an adapted formula which takes into account the variance of the estimations s^2_{ji} can be used to calculate the variance of the reported means:

$$s^2_{(j)} = \frac{s_j^{\,2}}{n_j} + \frac{s^2_{j1} + s^2_{j2} + \ldots + s^2_{jn}}{n_j^{\,2}} \qquad \textbf{(2-8)}$$

Equation 2-8 calculates an estimator of the variance of $\overline{\beta}_{(j)}$, with s^2_{ji} being the estimator of the variance of $\hat{\beta}_{ji}$ for repetition i and s^2_j the estimator of the sample variance of the n_j parameter estimates $\hat{\beta}_{ji}$ for one specific condition.

An adapted t-test was developed to estimate whether the parameters differ significantly between groups of experiments. The test statistic is calculated as

$$t_w = \left| \frac{\overline{\beta}_{(1)} - \overline{\beta}_{(2)}}{\sqrt{s^2_{(1)} + s^2_{(2)}}} \right| \qquad \textbf{(2-9)}$$

with $s^2_{(j)}$ calculated as in equation 2-8 and $\overline{\beta}_{(j)}$ given by

$$\overline{\beta}_{(j)} = \frac{\hat{\beta}_{j1} + \hat{\beta}_{j2} + \ldots + \hat{\beta}_{jn}}{n_j} \qquad \text{(j=1,2)} \qquad \textbf{(2-10)}$$

of which $\hat{\beta}_{ji}$ is the parameter value of repetition I (i=1, …, n).

The test statistic t_w is compared to the threshold value $t_{n_1+n_2-2,\ 0.05}$ under the student t distribution where n_1 is the number of repetitions for group1 and n_2 is the number of repetitions for group 2.

This recalculation of the standard deviations and the adapted t-test were applied on the data obtained from time sweep experiments (Chapter 4) and uniaxial extension (Chapter 5-6).

Chapter 3

Rotational rheometry for analyzing dough viscoelasticity

"iam pridem, ex quo suffragia nulli uendimus, effudit curas; nam qui dabat olim imperium, fasces, legiones, omnia, nunc se continet atque duas tantum res anxius optat, panem et circenses."

[Juvenalis, Satire X]

Relevant publication:

Van Bockstaele, F., De Leyn, I, Eeckhout, M. & Dewettinck, K. Non-linear creep-recovery measurements as a tool for evaluating the viscoelastic properties of wheat flour dough, Journal of Food Engineering, Submitted.

3.1. Problem statement

A rotational rheometer is the most frequently used type of equipment to determine the fundamental rheological properties of foods (Whorlow, 1992). Also in cereal science, rotational rheometry is a popular tool for determining dough rheological properties. However, before a measurement can be made, the dough sample has to be loaded between the parallel plates of the rheometer. During this loading procedure, the dough is compressed to reach a final gap width. The entire geometry should be filled with the dough sample. Thus, sample loading itself imposes stresses in the sample which should first be allowed to relax or decay before the actual measurement (dynamic oscillation or creep-recovery) is started. Faubion et al. (1985) already pointed out the importance of the resting period after sample loading because residual stresses in the sample can influence subsequent measurement. Because dough is highly sensitive to manipulation, a well considered sample loading protocol is crucial for obtaining reproducible data.

Once a dough sample is loaded and stresses caused by loading have decayed, a dynamic oscillation or creep-recovery experiment may be performed. In dynamic oscillation, the most used test types are a strain sweep, frequency sweep or time sweep experiment as described in 1.3.3.1.1.

In creep-recovery measurements, the dough sample is exposed to a constant shear stress which causes the sample to deform as explained in 1.3.3.1.2. Mostly, creep-recovery measurements are carried out in the linear viscoelastic region (LVR). However, creep-recovery can also be used to determine the non-linear behaviour of dough (Bloksma, 1962; Hibberd and Parker, 1979; Lefebvre, 2006; Lefebvre, 2009; Rouille et al., 2005). This may be interesting for bread dough because during processing, dough is subjected to strains and shear rates which fall beyond the linear domain (Lefebvre, 2006). Applying larger stresses (Khatkar and Schofield, 2002) or strains (Safari-Ardi and Phan-Thien, 1998) outside the LVR can be useful for predicting breadmaking potential. Non-linear creep also proved to be able to rank durum wheat cultivars according to expected dough strength (Edwards et al., 1999). However, studies concerning the non-linear behaviour of wheat flour dough in shear are limited and are usually performed at a single value of shear strain, stress or rate which are often arbitrarily chosen (Lefebvre and Mahmoudi, 2007).

Unlike dynamic oscillation tests, no clear rules exist on the amount of stress used in creep, the length of the creep phase and the length of the recovery phase in which the sample is able to recover from the creep deformation. As shown in Table 1.4 of section 1.3.3.1.2, a large variety in creep-recovery methodologies has been used in previous studies. Creep stresses varied from 0.3 till 1000 Pa, creep times varied from 60s till 3h and recovery times are situated between 100s and 12h. As it is the intention to use non-linear creep-recovery tests to evaluate dough rheological properties, it is necessary to first gain more insight in the effect of the creep-recovery methodology on the measured viscoelastic properties.

3.2. Research strategy

The first goal was to develop a dough loading protocol for the rheometer which allows effective sample loading while minimizing structure damage caused by sample compression. For this purpose, dough samples were compressed at different speed protocols and to different final gap set widths. The relaxation of the samples after compression was followed by starting a dynamic oscillation experiment which allows following up the normal force decay and the evolution of the dynamic moduli.

Secondly, creep-recovery methodology to measure non-linear viscoelastic properties in shear was investigated in more detail. The effect of recovery time (up to 3h), creep time (5-10-15-20min) and shear stress (10-100-250-500-1000Pa) on creep-recovery behaviour will be studied. By applying the Burgers model, more insight will be gained in the elastic, retarded elastic and viscous part of the observed deformation.

All measurements were performed on dough samples prepared of the same wheat flour (Bussard wheat flour).

3.3. Materials and methods

3.3.1. Wheat flour and dough preparation

Flour from wheat cultivar Bussard was obtained from Paniflower (Ghent, Belgium). Flour quality characteristics are summarized in Table 3.1.

Table 3.1 Bussard wheat flour properties

Protein content (%dm)	15.8	Farinograph*	WA (%)	63.7
Gluten index (%)	98.6		DDT (min)	7.4
Zeleny (mL)	70		Stability (min)	11.4
Wet gluten (%)	34.4	Alveograph**	P (mm H_20)	67.7
Ash content (%dm)	0.60		L (mm)	187.2
Damaged starch (%)	5.75		P/L	0.36
Falling number (s)	331		W (10^{-4} J)	311.4

*WA: water absorption; DDT: dough development time
** P: tenacity; L: extensibility; W: deformation energy

As can be seen in Table 3.1, the Bussard flour is a high quality flour with a high protein content and water absorption. Dough was prepared in an alveograph mixer bowl by mixing 250 g flour of 14% moisture with an amount of deionised water corresponding to 95% of the farinograph water absorption. The amount of water was slightly reduced to improve the manageability of the dough. Doughs were mixed for 5 minutes which resulted in a homogeneous, fully developed dough.

3.3.2. Rotational rheometry

3.3.2.1. Rheometer, geometry and sample loading

Dynamic oscillation and creep-recovery measurements were performed on an AR2000 controlled stress rheometer (TA Instruments, Brussels, Belgium). A serrated parallel plate geometry was used for all measurements (Figure 3.1).

Figure 3.1. AR2000 rheometer, Peltier element and serrated parallel plate geometry

The serrated plates were chosen to enhance the grip on the sample and to prevent wall slip during the measurements. The lower serrated plate was attached on a Peltier plate which assured accurate temperature control. The parallel plate system allows samples containing particles to be effectively measured as the gap can be positioned to any distance. The main disadvantage of a parallel plate system is that the stress is not uniform across the entire diameter. However, the software compensated for this and the shear stress and shear rate factors are given with respect to the rim.

3.3.2.2. Sample loading

The effect of the sample compression procedure (standard, linear, exponential and NF control) and the gap set width (1000µm, 2000µm and 3000µm) on sample relaxation and dynamic moduli was investigated. A time sweep experiment was started after sample loading with an applied strain of 0.01% at a frequency of 1Hz. The chosen strain was below the limit of the linear viscoelastic region (0.1%). Separate measurements were always performed on freshly mixed dough.

3.3.2.3. Creep-recovery experiments

First, the appropriate model was selected to describe the creep-recovery data. To decide whether to use the four or six parameter Burgers model to describe the creep data and the three or five parameter model to describe the recovery data, the statistical F-test was used as described in 2.2.7.1. The test statistics were calculated for three 'reference' creep-recovery curves with creep phases of 5 min at 100 Pa, 20 min at 100 Pa and 5 min at 500 Pa. All recovery phases lasted for 10 minutes.

The 4- and 6-parameter Burgers models for describing the creep data are given by:

$$J = f(t) = J_0 + J_1\left(1 - exp\left(\frac{-t}{r_1}\right)\right) + \frac{t}{\mu_0} \quad \textbf{(3-1)}$$

$$J = f(t) = J_0 + J_1\left(1 - exp\left(\frac{-t}{r_1}\right)\right) + J_2\left(1 - exp\left(\frac{-t}{r_2}\right)\right) + \frac{t}{\mu_0} \quad \textbf{(3-2)}$$

The 3- and 5-parameter models for describing the recovery data are given by:

$$J = f(t) = J_0 + J_1\left(1 - exp\left(\frac{-t}{r_1}\right)\right) \quad \textbf{(3-3)}$$

$$J = f(t) = J_0 + J_1\left(1 - exp\left(\frac{-t}{r_1}\right)\right) + J_2\left(1 - exp\left(\frac{-t}{r_2}\right)\right) \quad \textbf{(3-4)}$$

Secondly, the amount of recovery time necessary to evaluate the elastic recovery of the dough after creep deformation was determined. Two experiments are shown in which the recovery was recorded during 3 hours after a creep phase of 5 min at shear stress of 100 or 500Pa. The recovery compliance after 10 min was compared to the recovery compliance obtained after 1, 2 and 3 hours.

Further, to investigate the effect of creep time, a shear stress of 100 Pa was set on Bussard dough samples for 5, 10, 15 and 20 minutes. This creep phase was always followed by a recovery phase of 10 min. At least three independent replicates were analyzed, always representing a freshly mixed dough.

Finally, Bussard dough samples were also subjected to shear stresses of 10, 100, 250, 500 and 1000 Pa during 5 min. This creep phase was followed by a recovery phase of 10 min. At least three independent replicates were analyzed, always representing a freshly mixed dough.

3.4. Results and discussion

3.4.1. Development of sample loading protocol

3.4.1.1. Normal force

The normal force (NF) [N] is the force acting in the direction of the bob shaft of the measuring system, trying to push the upper plate or cone upwards (and the lower plate downwards). The rheometer measures the NF in the axial direction (y-direction) (Mezger, 2002). During sample compression the normal force increases to reach a maximum as the final gap position is reached. From this point on, the sample will relax and the NF will decay (Figure 3.2). The NF decreases to reach an equilibrium value after a certain period of time. To

follow up the normal force after sample loading, a time sweep experiment can be started as is shown in Figure 3.2. The time sweep itself does not cause structural damage so the measurement only reflects changes due to sample relaxation. Both G' and the NF decrease to reach a relatively constant level.

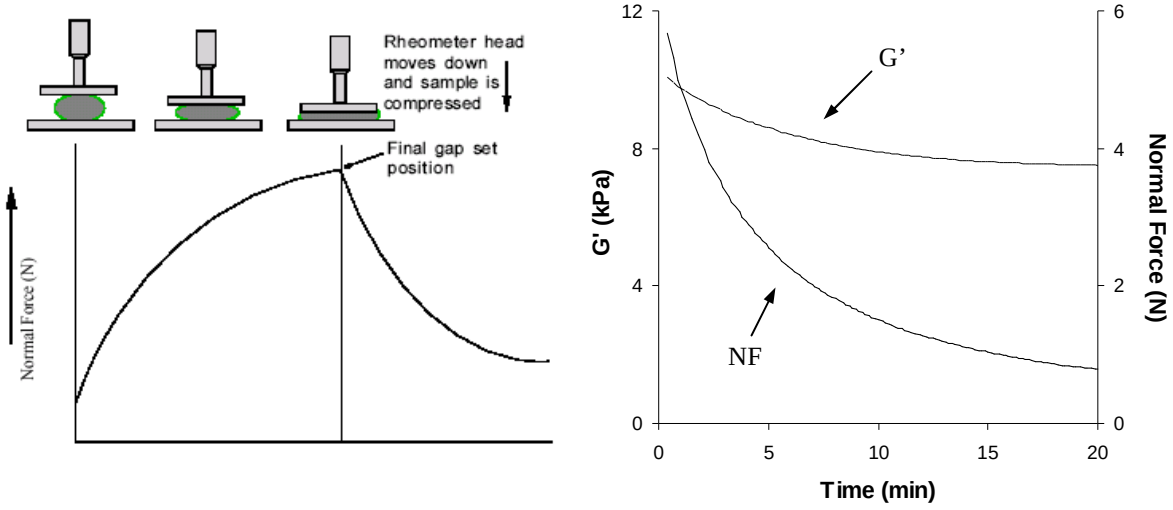

Figure 3.2 Evolution of normal force during and after sample loading between parallel plates (From TA Instruments) and an example of a dynamic oscillation time sweep experiment in which the evolution of normal force (NF) and elastic modulus (G') is followed as function of time

3.4.1.2. Sample compression

A biaxial pre-orientation of the dough structures is always obtained during sample loading. Depending on the structure and the cohesion of the dough, the pre-orientation may have a large destructuring effect (Davidou et al., 2008).

The rheometer provides four options for sample compression: standard (1000 µm/s), linear (other constant speed), exponential (slower compression when final gap position is almost reached) and max. NF (compression until preset NF is reached, compression is only continued as NF decays below preset NF). The exponential and max. NF options were found to be very impractical as the final gap setting was reached very slowly. In the meantime, dough samples were exposed to the air and they started to dehydrate. Therefore it was decided to compress dough samples at a constant but lower speed than the standard option, more specifically at 500 µm/s. This guaranteed a minimal sample dehydration during loading.

3.4.1.3. Final gap position

To select the final gap position, an experiment was set up in which dough samples were compressed at a speed of 500 µm/s to gap widths of 1000, 2000 or 3000 µm. The evolution of the normal force after sample loading is shown in Figure 3.3. The highest normal force values were obtained for a gap of 1000 µm. Also a high residual normal force can be observed after 20 min indicating high residual stresses in the sample. Although only small normal force values were found when applying a gap width of 3000 µm, it was also observed that at this gap width, samples were more sensitive for dehydration during the measurements. So ultimately, a gap width of 2000 µm was selected.

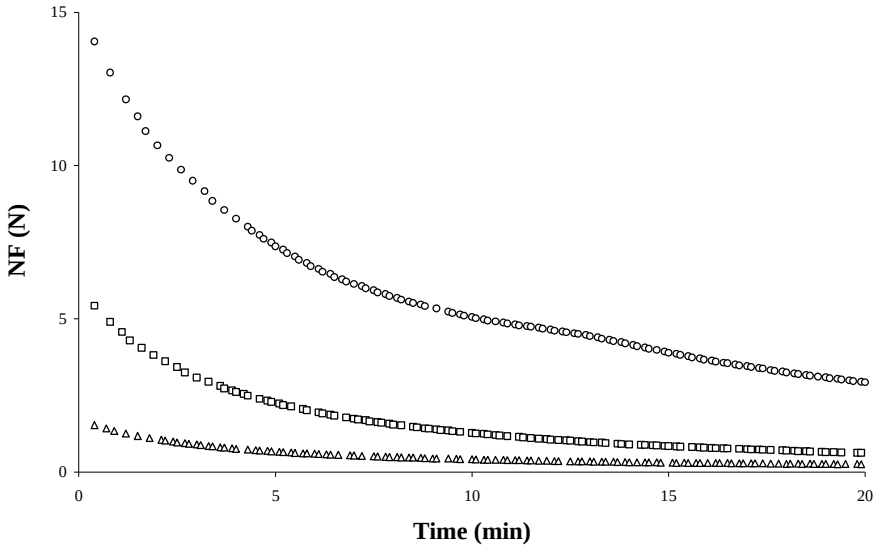

Figure 3.3 Evolution of normal force (NF) after compressing dough samples at 500 µm/s to reach a gap width of 1000 (○), 2000 (□) or 3000 (Δ) µm

3.4.1.4. Sample loading protocol

The sample loading protocol was developed to minimize structural damage and sample dehydration during loading. A dough piece with a mass of approximately 7 g was taken from the inner side of a dough and manipulated as little as possible. The dough piece was transferred to the rheometer and immediately compressed between the serrated parallel plates at a speed of 500 µm/s to obtain a gap of 2000 µm. The excess dough was removed to obtain a proper filling of the geometry. Water drops were placed around the dough and in the water reservoir on top of the geometry. A solvent trap was placed around the geometry to prevent sample dehydration by creating a saturated water atmosphere. A schematic drawing of the

setup of the parallel plate geometry for analyzing dough rheology with rotational rheometry is shown in Figure 3.4.

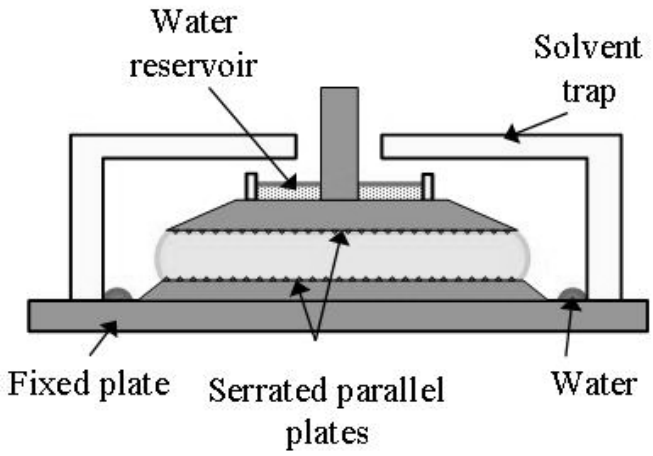

Figure 3.4 Schematic drawing of the rheometer parallel plate geometry with positioning of the solvent trap

In literature a lot of variation exists concerning the amount of time required for dough relaxation before a dynamic or transient test is started. Dough resting times after sample loading vary from 1 (Lindahl and Eliasson, 1992) to 60 minutes (Baltsavias et al., 1997). However, when applying a fixed relaxation time, dough samples could not have been fully relaxed at the time the subsequent measurement is started. Autio et al. (2001) used a fixed normal force (0.4 N) at the start of the measurement for all dough samples but this was found impractical as some dough samples would only reach this value after very long resting times. Therefore, it was decided to monitor the normal force for each dough individually by starting a time sweep experiment after sample loading. The time sweep is performed at a strain of 0.01% and a frequency of 1 Hz. This guarantees deformation conditions inside the LVR. During this experiment the normal force is monitored and if the normal force reaches an equilibrium, another test (dynamic oscillation or creep-recovery) can be started. From a practical point of view, it was stated that NF reached equilibrium conditions if the last 20 data points of NF did not show a deviation higher than 2%. This allowed sample relaxation over reasonable time lengths.

3.4.2. Creep-recovery methodology

3.4.2.1. Model selection

In Table 1.4 of section 1.3.3.1.2 it was already shown that the 4- and 6-parameter Burgers models are frequently used to model creep data. A statistical F-test is used to decide whether the addition of an extra Kelvin-Voigt element to the 4-parameter model significantly improves the fit to the original data. To find the best model to describe the creep-recovery data, three 'reference' creep-recovery curves for Bussard flour-water dough differing in creep time and applied shear stress were selected to which the different models were fitted (Figure 3.5).

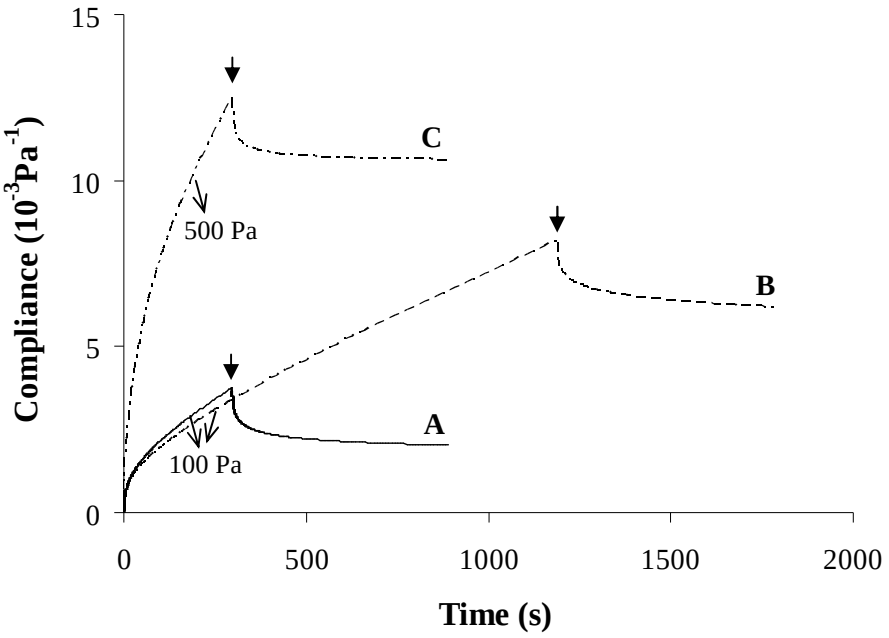

Figure 3.5 Three example creep-recovery curves for Bussard flour-water dough obtained at different creep times and shear stresses during the creep phase: [A] shear stress 100 Pa during 5 min; [B] shear stress 100 Pa during 20 min; [C] shear stress 500 Pa during 5 min. An arrow indicates the point of release of the shear stress after which the recovery is recorded during 10 min.

The models were fitted to the data and the obtained SSR values and the calculated F statistic are summarized in Table 3.2. For all the 'reference' creep-recovery curves the more complicated model was found to describe the data significantly better as $F_{calculated} > F_{tabulated}$. The more complex model caused a clear decrease in SSR values indicating better fit of the original data. The 6- and 5-parameter model will thus be used in the further research to fit the

creep-recovery data. Figure 3.6 illustrates the ability of the different models to describe the original data.

Table 3.2 Model selection based on a statistical F-test

Curve		*A*	*B*	*C*
Creep	SSR (4p-model)	2.95E-06	7.06E-06	1.39E-05
	SSR (6p-model)	3.18E-07	8.31E-07	1.33E-06
	$F_{calculated}$	2101	2200	2396
	$F_{tabulated}$	3.01	3.01	3.01
	Model selected	6p	6p	6p
Recovery	SSR (3p-model)	8.56E-06	9.75E-06	7.70E-06
	SSR (5p-model)	9.83E-07	1.02E-06	9.40E-07
	$F_{calculated}$	2112	2341	1970
	$F_{tabulated}$	3.01	3.01	3.01
	Model selected	5p	5p	5p

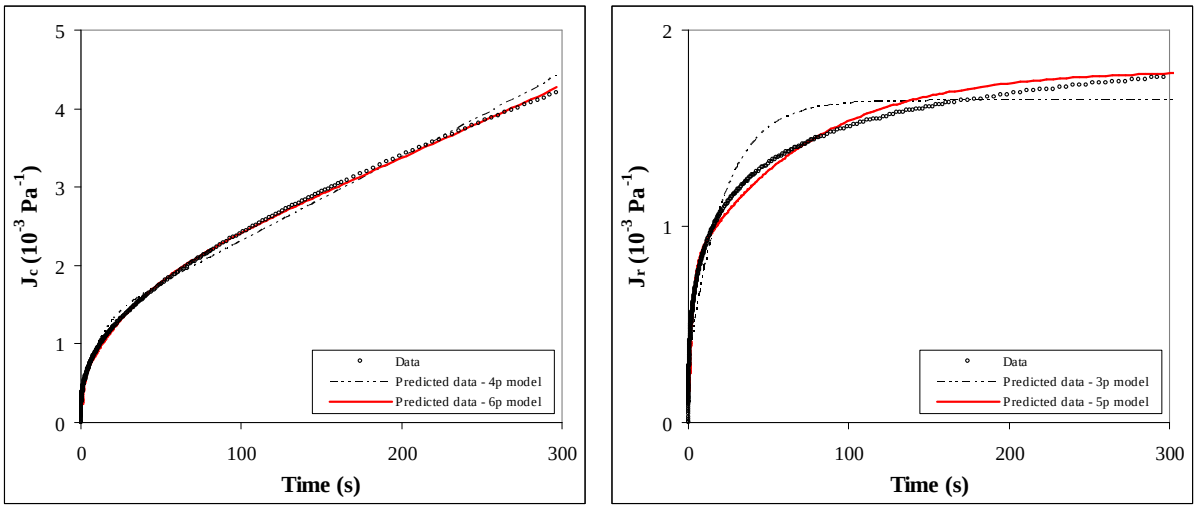

Figure 3.6 Predicted values obtained by application of the different Burgers models in comparison with the original data for describing the creep (J_c) or recovery (J_r) compliance (data correspond to reference curve A).

3.4.2.2. Determining the required recovery time

In literature, recovery times have been reported from 60s up to 12h (Table 1.4 from section 1.3.3.1.2). A recovery time equivalent to the creep time seems to be the minimum and in most cases the length of the creep phase is two or three times that of the creep phase.

To decide on the appropriate length of the recovery phase two creep experiments (5 min creep with a shear stress of 100 and 500 Pa) were followed by a recovery phase which lasted for 3

hours. The amount of recoverable compliance recorded after 10 min recovery was compared to the recoverable compliance found after 1, 2 and 3 hours of recovery (Table 3.3).

Table 3.3 Recoverable compliance (J_r) as function of increasing recovery time after a creep time of 5 min at a shear stress of 100 and 500 Pa

Recovery time (min)	**J_r (100 Pa)** (10^{-3} Pa^{-1})	**J_r (500 Pa)** (10^{-3} Pa^{-1})
10	2.06	1.92
60	2.29	1.87
120	2.33	1.79
180	2.35	1.73

For the creep experiment at 100 Pa, recoverable compliance obtained after 10 min represents 90 and 88% of the total recoverable compliance recorded after 1 and 3 hours respectively. So, most of the recovery is observed in the first 10 min. For the experiment at a shear stress of 500 Pa, surprisingly, a decrease was found for the recoverable compliance with increasing recovery time. So, the maximum value for recoverable compliance could be obtained after 10 min, after which part of the recovered compliance is lost again. The reason for this is not fully clear. Applying such a high shear stress, induces a large shear strain (up to 700%) which is thought to damage dough structure in such a way that elasticity is lost.

A recovery time of 10 min was thus found to be sufficient to detect most of the elastic recovery of the dough sample and was applied in the further research.

3.4.2.3. Effect of creep time

Creep-recovery curves of Bussard flour-water dough were recorded at different creep times ranging from 5 to 20 min. Two example curves (A-B) are shown in Figure 3.5. The purpose was to investigate when the dough reaches steady state deformation during creep. Steady state deformation is reached when the creep deformation is mainly governed by viscous flow which is seen as a linear increase of the creep curve. Also, the viscous flow deformation cannot be recovered in the recovery phase. For this purpose the maximum creep compliance ($J_{c,max}$) and the maximum recovery compliance ($J_{r,max}$) obtained at the end of the creep and recovery phase, respectively, were measured and compared.

The effect of increasing creep time on $J_{c,max}$ and $J_{r,max}$, for Bussard flour-water dough put under a shear stress of 100 Pa, is presented in Figure 3.7.

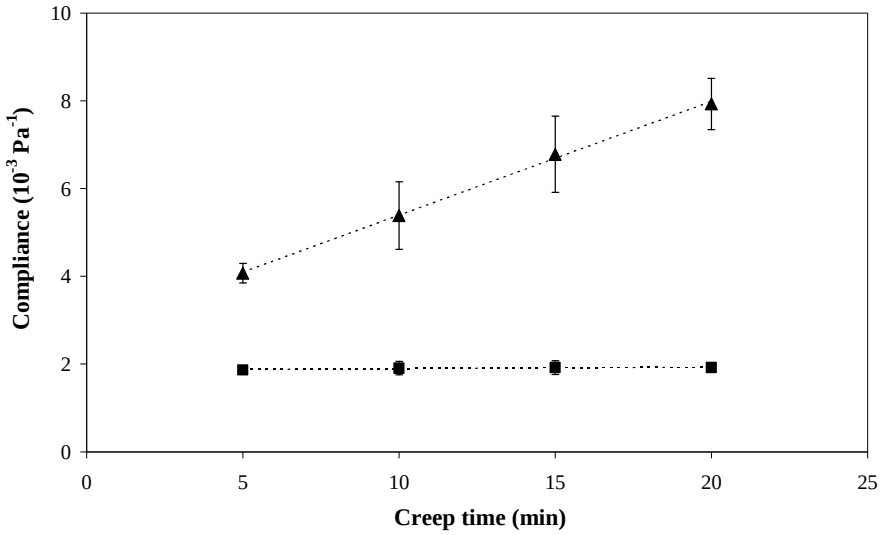

Figure 3.7 Maximum creep compliance ($J_{c,max}$; ▲) and maximum recovery compliance ($J_{r,max}$; ■) obtained at the end of the creep and recovery phase respectively, as function of the applied creep time at a shear stress of 100 Pa for Bussard flour-water dough. $J_{r,max}$ recorded 10 min after removal of the shear stress. Error bars indicate standard deviation.

It can be seen that $J_{c,max}$ increases linearly with increasing creep time which suggests that steady state shear conditions are already reached at a creep time as short as 5 min. The extra deformation obtained for longer creep times, can be mainly attributed to viscous flow. Similar results have already been reported for durum wheat doughs (Edwards et al., 1999) and biscuit doughs (Pedersen et al., 2004). On the other hand, Lefebvre (2006) stated that steady state in creep requires several hours to be established.

It is also shown that an increase in creep time does not affect the amount of recoverable compliance observed during the recovery phase. This means that the elastic response (instantaneous and retarded) is not changed when the dough is put under this stress for a longer time and the extra deformation obtained after longer creep time can be assigned as viscous deformation which is not recoverable.

A creep time of 5 minutes was selected for the further research as steady state conditions were shown to be reached at that creep time.

To get more insight in the effect of creep time on the instantaneous elastic, retarded elastic and viscous deformation, the six-parameter Burgers model was applied to the creep-recovery

curves (Table 3.4). Increasing the creep time, has a pronounced effect on the modeled parameters of the creep phase. Especially the retardation times and steady state viscosity μ_0 are affected. Compliances J_0, J_1 and J_2 only slightly increase due to longer creep times. Edwards et al. (2003) also noted that the terms from the Burgers model are a function of time and only reach a steady-state after long time. So, in contrast to the linear increase of $J_{c,max}$ which indicates steady state, the parameters of the Burgers model extracted from the creep phase are not yet constant. Tronsmo et al. (2003a) already showed that although steady-state may not be reached completely and the obtained parameters like steady-state viscosity may not be the true fundamental values, they may be useful in distinguishing between different samples.

Table 3.4 Effect of creep time on Burgers model parameters for Bussard dough at a shear stress of 100 Pa[*,]**

Creep time	J_0 (10^{-4} Pa^{-1})	J_1 (10^{-4} Pa^{-1})	J_2 (10^{-3} Pa^{-1})	r_1 (s)	r_2 (s)	μ_0 (10^5 Pa.s)
Creep phase						
5	1.27 ± 0.05^{a}	4.12 ± 0.17^{a}	0.92 ± 0.07^{a}	1.14 ± 0.08^{a}	29.02 ± 1.79^{a}	1.11 ± 0.06^{a}
10	1.42 ± 0.07^{b}	$4.60 \pm 0.48^{a,b}$	0.97 ± 0.12^{a}	1.66 ± 0.11^{b}	51.01 ± 2.37^{b}	1.56 ± 0.25^{b}
15	1.49 ± 0.02^{b}	4.93 ± 0.46^{b}	$1.06 \pm 0.11^{a,b}$	2.18 ± 0.09^{c}	72.29 ± 2.19^{c}	$1.61 \pm 0.12^{b,c}$
20	1.52 ± 0.02^{b}	5.30 ± 0.18^{b}	1.19 ± 0.06^{b}	2.93 ± 0.02^{d}	105.9 ± 1.11^{d}	1.94 ± 0.17^{c}
Recovery phase[***]						
5	1.69 ± 0.07^{a}	6.28 ± 0.28^{a}	0.99 ± 0.03^{a}	2.43 ± 0.12^{a}	71.92 ± 1.80^{a}	
10	1.73 ± 0.08^{a}	6.22 ± 0.51^{a}	1.01 ± 0.09^{a}	2.63 ± 0.12^{a}	89.78 ± 1.89^{b}	
15	1.72 ± 0.03^{a}	6.34 ± 0.19^{a}	1.08 ± 0.03^{a}	2.85 ± 0.11^{b}	100.27 ± 1.47^{c}	
20	1.65 ± 0.05^{a}	6.00 ± 0.19^{a}	1.06 ± 0.04^{a}	2.97 ± 0.07^{b}	107.37 ± 3.19^{d}	

[*] J_0: instantaneous compliance; J_1, J_2: retarded elastic compliances; r_1, r_2: retardation times; μ_0: steady state viscosity
[**] Mean and standard deviation based on at least three repetitions. Data in the same column for creep or recovery phase, indicated with a different letter are significantly different (α=0.05)
[***] Recovery recorded during 10 min after the end of the creep phase

For the recovery phase, no significant changes in compliance values (J_0, J_1 and J_2) occur but the retardation times, especially r_2, show an increase with increasing creep time. It seems that creep time does not affect the amount of elastic recovery but influences the speed of the recovery since higher retardation times indicate a slower retarded elastic response. To illustrate this phenomenon, the normalized recovery curves are shown in Figure 3.8. This indicates that by extending the creep time, elastic bonds are stretched further but are not

broken as this would negatively affect total recovery. Longer creep times will cause a slower elastic response of the sample.

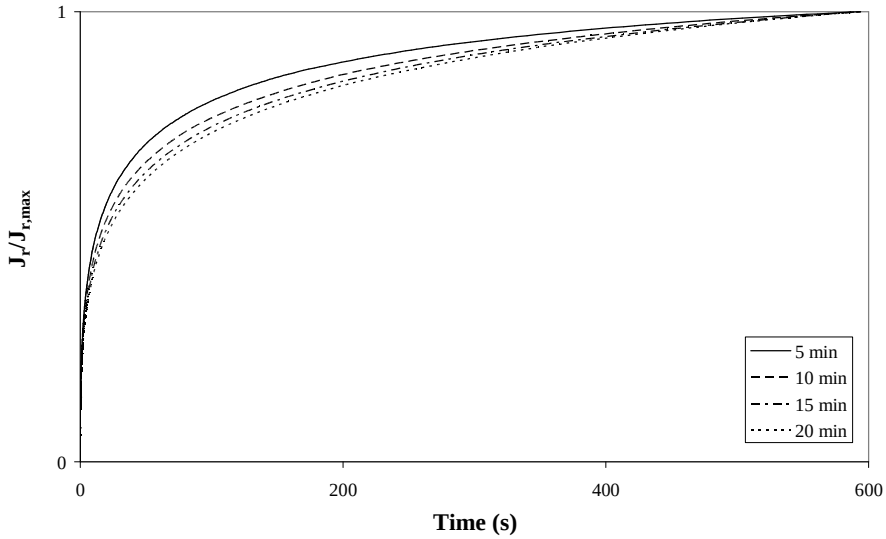

Figure 3.8 Normalized recovery compliance curves ($J_r/J_{r,max}$) for doughs that have been subjected to a shear stress of 100 Pa during different creep times

3.4.2.4. Effect of shear stress

The effect of different shear stresses (10, 100, 250, 500 and 1000 Pa) on the creep-recovery deformation was investigated. A creep and recovery phase of 5 and 10 min respectively was used as previously determined. Figure 3.9 shows the creep-recovery curves performed at different shear stresses during the creep phase. As the compliance increases with increasing shear stress, it is obvious that the measurements are made outside the LVR. Measurements made between 0.1 and 10 Pa provided evidence that 10 Pa was still situated inside the LVR. $J_{c,max}$ increases linearly with the applied shear stress up to 500 Pa after which a plateau seems to be reached.

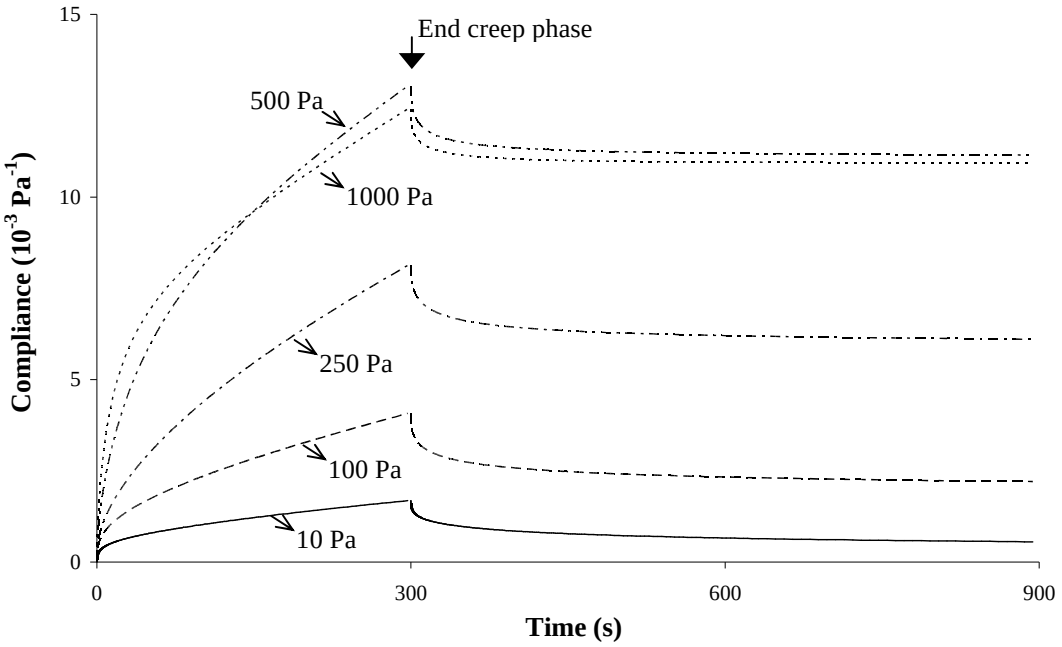

Figure 3.9 Creep-recovery curves of Bussard flour-water dough performed at different shear stresses (10Pa [−], 100Pa [− −], 250Pa[− · −], 500Pa[− · · −], 1000Pa[· · ·]). Curves are presented as the mean of at least three independent measurements.

The Burgers model was also fitted to the creep-recovery curves obtained at the different shear stresses. The results are summarized in Table 3.5. Shear stress clearly influences the shape of the creep curves which is also reflected in the model parameters since J_0, J_1, J_2 and r_1 increase with increasing shear stress. Retardation time r_2 shows an increase up to 250 Pa and then decreases again. Steady state viscosity μ_0 decreases with increasing shear stress and reaches a constant value at a shear stress of 250 Pa. Increasing the shear stress, induces a higher deformation of the dough sample which results in a lower dough viscosity.

In the recovery phase, $J_{r,max}$ increases first to reach a plateau between 100 and 500 Pa and then decreases again when a stress of 1000 Pa is applied. The plateau in total recovery between 100 and 500 Pa is also seen in the model parameters as J_0, J_1, J_2 stay relatively constant. For the retardation times r_1 and r_2, a decrease is observed with increasing shear stress. This means that retarded elastic recovery takes place faster when the sample is subjected to a higher shear stress in creep. The effect of shear stress on the elastic response in recovery is also presented in Figure 3.10. The curves are shifted to higher values due to the faster elastic response.

Table 3.5 Effect of shear stress on Burgers model parameters[*,]**

Shear stress	J_{max} (10^{-3} Pa^{-1})	J_0 (10^{-4} Pa^{-1})	J_1 (10^{-4} Pa^{-1})	J_2 (10^{-3} Pa^{-1})	r_1 (s)	r_2 (s)	μ_0 (10^5 Pa.s)
Creep phase							
10	1.68 ± 0.13^{a}	0.88 ± 0.03^{a}	$1.95 \pm 0.08^{a,d}$	0.39 ± 0.04^{a}	0.73 ± 0.06^{a}	23.69 ± 1.78^{a}	2.87 ± 0.26^{a}
100	4.07 ± 0.22^{b}	$1.27 \pm 0.05^{b,d}$	$4.12 \pm 0.17^{b,d}$	0.92 ± 0.07^{b}	1.14 ± 0.08^{b}	29.02 ± 1.79^{b}	1.11 ± 0.06^{b}
250	8.12 ± 0.13^{c}	$1.49 \pm 0.04^{c,d}$	$5.63 \pm 0.15^{c,d}$	$1.98 \pm 0.06^{c,d}$	1.43 ± 0.02^{c}	42.58 ± 0.28^{c}	0.54 ± 0.01^{c}
500	13.69 ± 1.17^{d}	2.00 ± 0.20^{d}	9.17 ± 1.86^{d}	4.72 ± 0.61^{d}	2.12 ± 0.12^{d}	40.28 ± 2.50^{c}	0.44 ± 0.04^{c}
1000	12.41 ± 0.33^{d}	$2.29 \pm 0.03^{e,d}$	18.08 ± 1.04^{e}	$4.47 \pm 0.12^{e,d}$	2.73 ± 0.19^{e}	24.28 ± 1.46^{a}	0.49 ± 0.03^{c}
Recovery phase[***]							
10	1.12 ± 0.06^{a}	1.23 ± 0.02^{a}	3.10 ± 0.18^{a}	0.64 ± 0.05^{a}	2.46 ± 0.23^{a}	98.74 ± 5.64^{a}	
100	1.87 ± 0.09^{b}	1.69 ± 0.07^{b}	6.28 ± 0.28^{b}	$0.99 \pm 0.03^{b,c}$	2.43 ± 0.12^{a}	71.92 ± 1.80^{b}	
250	2.02 ± 0.05^{b}	1.69 ± 0.03^{b}	6.85 ± 0.18^{b}	1.07 ± 0.03^{b}	2.27 ± 0.02^{a}	56.33 ± 0.52^{c}	
500	1.95 ± 0.11^{b}	$1.68 \pm 0.08^{b,c}$	6.41 ± 0.40^{b}	0.95 ± 0.06^{c}	$1.62 \pm 0.08^{b,c}$	37.25 ± 3.26^{d}	
1000	1.47 ± 0.01^{c}	1.54 ± 0.03^{c}	5.22 ± 0.02^{c}	0.75 ± 0.02^{d}	1.19 ± 0.13^{c}	29.33 ± 3.76^{d}	

[*] J_{max}: maximum compliance in creep or recovery phase; J_0: instantaneous compliance; J_1, J_2: retarded elastic compliances; r_1, r_2: retardation times; μ_0: steady state viscosity

[**] Mean and standard deviation based on at least three repetitions. Data in the same column for creep or recovery phase, indicated with a different letter are significantly different (α=0.05)

[***] Recovery recorded during 10 min after the end of the creep phase

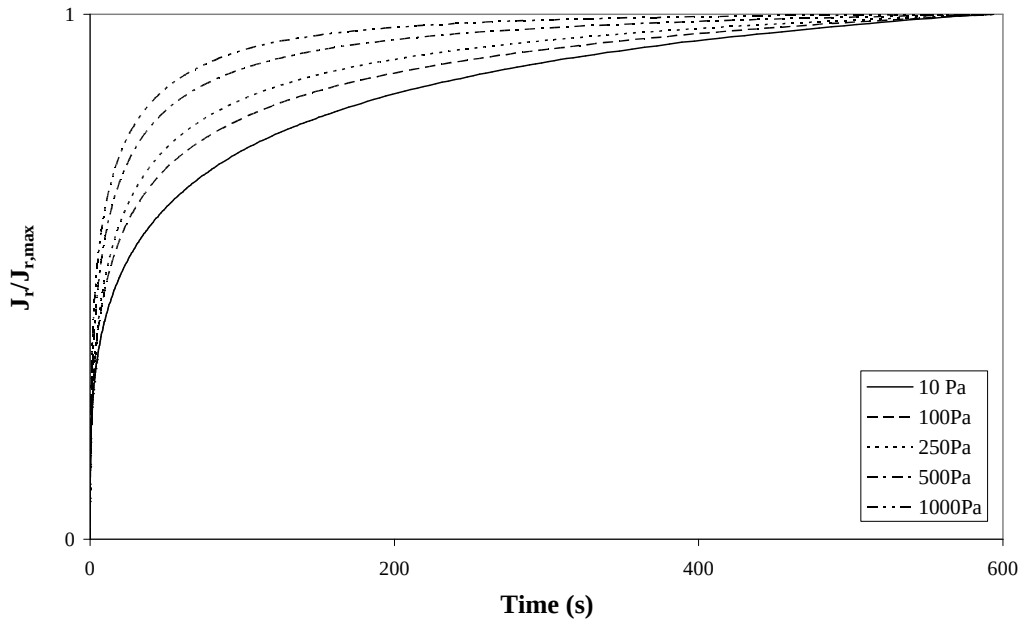

Figure 3.10 Normalized recovery compliance curves ($J_r/J_{r,max}$) for doughs subjected to different shear stresses during a creep time of 5 min

3.5. Conclusions

In this chapter, first the importance of a well standardized sample loading protocol was discussed for the application of rotational rheometry to wheat flour dough. A sample loading protocol was developed with the aim of minimizing sample dehydration and structural breakdown during loading. Eventually it was chosen to compress dough samples with a linear speed of 500 µm/s to a gap of 2000 µm. By placing a solvent trap and creating a saturated water atmosphere it was possible to prevent sample dehydration. It was shown how sample relaxation can be monitored by using a small deformation time sweep experiment by which the normal force decay can be monitored. After sufficient relaxation, a dynamic oscillation or a creep-recovery experiment may be started.

In the second part of this chapter, the creep-recovery methodology was investigated in more detail. The 6- and 5-parameter Burgers models showed a significant better fit to the creep and recovery data, respectively. Further, a recovery time of 10 minutes was found to be sufficient to obtain most of the recovery after the creep deformation. It was also observed that a creep time of 5 minutes was sufficient to reach (pseudo) steady state conditions in creep. Shear stress was shown to strongly influence the creep response but total recovery was less influenced. The recovery retardation times, on the other hand, were very sensitive to changes in shear stress. It was also observed that shear stresses of 500 or 1000 Pa induced large strains in the sample, possibly damaging the elastic response of the sample.

To summarize, for further experiments creep-recovery measurements will be performed with a creep phase of 5 min followed by a recovery phase of 10 min. In Chapter 4, where this methodology is applied, shear stresses of 100 and 250 Pa were chosen to determine the non-linear viscoelastic dough properties.

Chapter 4

Rheological properties of wheat flour dough and the relationship with bread volume

GEBIEREN: witte korstjes van brood, waar de broden in de oven tegen elkaar gelegen hebben.

[Oilsjtersen Diksjoneir]

Relevant publications:

Van Bockstaele, F., De Leyn, I, Eeckhout, M. & Dewettinck, K. (2008). Rheological properties of wheat flour dough and the relationship with bread volume. I. Creep-recovery measurements. Cereal Chemistry, 85(6), 753-761.

Van Bockstaele, F., De Leyn, I, Eeckhout, M. & Dewettinck, K. (2008). Rheological properties of wheat flour dough and the relationship with bread volume. II. Dynamic oscillation measurements. Cereal Chemistry, 85(6), 762-768.

Van Bockstaele, F., De Leyn, I, Eeckhout, M. & Dewettinck, K. Non-linear creep-recovery measurements as a tool for evaluating the viscoelastic properties of wheat flour dough, Journal of Food Engineering, Submitted.

4.1. Introduction

Rotational rheometry is one of the most popular and widely used fundamental rheological techniques for measuring the viscoelastic properties of cereal doughs and batters (Dobraszczyk and Morgenstern, 2003). Dynamic rheological testing has become a powerful and preferred tool for examining the structure and the fundamental properties of wheat flour doughs (Song and Zheng, 2007). Next to dynamic tests, also creep-recovery measurements are being increasingly used to analyze dough viscoelasticity. However, up till now rheological parameters of wheat flour dough obtained by rotational rheometry have not been successfully related to the breadmaking potential of wheat flour.

In case of the dynamic tests, still a lot of debate is going on whether these tests are appropriate in predicting end-use quality. Many authors have reported a lower value for the elastic modulus G' for the higher quality baking flour (Autio et al., 2001; He and Hoseney, 1991; Khatkar and Schofield, 2002). In contrast, others reported that a higher value for G' and a lower value for the phase angle δ were related to better breadmaking performance (Abdelrahman and Spies, 1986; Janssen et al., 1996a; Navickis et al., 1982). In other studies no difference in elastic modulus G' was observed for bread doughs (Hayman et al., 1998; Safari-Ardi and Phan-Thien, 1998; Sliwinski et al., 2004a) and biscuit doughs (Pedersen et al., 2004) from different wheat cultivars. Until now, better results have been obtained by studying the gluten instead of the more complex dough system. Gluten from high quality baking flours showed higher values for G' (He and Hoseney, 1991; Janssen et al., 1996b; Khatkar et al., 1995; Khatkar and Schofield, 2002; Kokelaar et al., 1996). Sliwinski et al. (2004c) who reported differences in dynamic parameters for gluten of various cultivars, were not able to rank the cultivars according to their breadmaking potential.

Creep-recovery has been used to differentiate between wheat flour glutens (Tronsmo et al., 2003b) and durum wheat dough samples (Edwards et al., 1999) of varying quality. Very low correlations were obtained between bread volume and creep-recovery parameters for gluten (Tronsmo et al., 2003c). Wang and Sun (2002) investigated the rheological properties of eleven commercial wheat flours by creep-recovery tests performed in compression. They found a high correlation between maximum recovery strain and bread volume. Creep-recovery experiments have also been performed on undeveloped dough samples but no

correlations with the bread volume have been found (Campos et al., 1997; Stojceska et al., 2007).

4.2. Objective

Wheat flour is a raw material of which the complex chemical composition is determined by wheat cultivar, cultivation conditions and processing. Many quality tests and parameters have been developed to assess wheat flour quality and functionality. Among those tests, determining dough rheological properties is a popular way for predicting the functionality of wheat flour in the breadmaking process.

The aim of this chapter is to analyze the rheological properties of wheat flour dough from different wheat samples by means of rotational rheometry (dynamic oscillation and creep-recovery) and to correlate the obtained parameters to the breadmaking potential of wheat flour, as shown by bread volume.

4.3. Research strategy

Wheat flours from 17 pure wheat cultivars were analyzed for a set of standard quality parameters, rheological properties and baking quality. Standard quality evaluation included determination of protein and ash content, damaged starch, wet gluten, Zeleny sedimentation value and Hagberg falling number. Further, high molecular weight glutenin subunit (HMW-GS) composition of the wheat flours was determined.

Rheological properties of the wheat flour doughs were determined by means of empirical methods (farinograph and alveograph) and rotational rheometry. Rotational rheometry provided information about the linear viscoelastic properties (dynamic oscillation time sweep) and the non-linear viscoelastic properties (creep-recovery). The 6-parameter Burgers model was used to model the creep-recovery curves. For rotational rheometry, flour-water doughs were prepared with a water content proportional to the farinograph water absorption to obtain similar dough consistencies, as applied in the breadmaking procedure.

An extensive correlation analysis was performed among the standard quality parameters and those obtained from rheology. Multiple linear regression with a forward model selection was

used to combine parameters which together may give a better prediction of the bread volume. Principal component analysis was used to visualize variability in the dataset.

4.4. Materials and methods

4.4.1. Wheat flour

A set of seventeen European wheat cultivars which are commercially available was used. Sixteen wheat samples were harvested at the experimental farm of University College Ghent. Flour of one cultivar (Bussard) was obtained from Paniflower (Ghent, Belgium). The wheat cultivars used in this study belong to four wheat classes (E, A, B and C) according to the German wheat classification (Table 4.1).

4.4.2. Rotational rheometry

The doughs for rheological testing were prepared in the mixing bowl of an alveograph (5 min mixing). The dough formula consisted of flour (250 g - 14% moisture) and deionised water (95% of farinograph water absorption). After mixing, the dough rested 10 min in a closed container, at room temperature. Sample loading was executed according to the protocol as described in 3.4.1. After sample loading, a time sweep oscillation experiment (0.01% - 1Hz) was started. During the time sweep, the dynamic moduli (G', G" and $|G^*|$) and phase angle δ were recorded. Sample relaxation was evaluated as described in 3.4.1. Values for the oscillation parameters were obtained from the last five data points of the time sweep measurement, of which the mean value was calculated. Standard deviations were recalculated according to 2.2.7.6. When the normal force had reached equilibrium conditions, a creep measurement was performed on the same dough piece at a shear stress of 100 or 250 Pa for 5 min, followed by a recovery phase of 10 min (shear stress = 0 Pa). Creep measurements of 100 and 250 Pa were performed on separate doughs. Reported values are themselves the mean of at least three independent replicates (separately mixed doughs). All measurements were performed at 20°C.

4.4.3. Breadmaking test

The breadmaking potential of the wheat flours was determined by a large scale breadmaking test as described in 2.2.5.1. This test was useful to determine the ability of the dough to retain the gas during breadmaking. The obtained bread volume is used as the indicator for breadmaking potential of the wheat flours.

4.5. Results and discussion

4.5.1. Flour quality

Moisture content of the flours after milling varied from 12.5 to 14% on dry matter. The results for protein and ash content, damaged starch, Zeleny sedimentation value, wet gluten, Hagberg falling number and baking volume for the flour of the 17 wheat cultivars are listed in Table 4.1. Further, Table 4.1 also contains information about the wheat class according to the German wheat classification and the HMW-GS composition of the wheat flours. The wheat flours show different quality as following ranges were observed for protein content (11.6-16.9%dm), ash content (0.56-0.90%dm), damaged starch (3.61-6.32%), Zeleny sedimentation value (34-70), wet gluten (20.7-37.2%), Glu-1 score (5-10) and Hagberg falling number (331-487) and baking volume (429-641cm^3/100g flour).

To better understand the relation between different quality parameters, a correlation analysis was conducted. Several correlations were found between the standard quality parameters. Protein content significantly correlated with wet gluten ($r=0.885^{**}$), Zeleny sedimentation value ($r=0.757^{**}$) and ash content ($r=0.617^{**}$). A similar correlation coefficient between protein content and Zeleny sedimentation value has been reported by Faergestad et al. (2000). In general, the amount of protein is highly correlated with the wet gluten content as around 80% of the wheat proteins are gluten proteins. Moreover, as protein content rises, also the relative amount of the gluten proteins in total protein content increases (Hoseney, 1992). Wet gluten is correlated with Zeleny sedimentation value ($r=0.692^{**}$). Ash content is also related with the falling number ($r=0.758^{**}$) and the amount of damaged starch correlates with the Zeleny sedimentation value ($r=0.572^{*}$).

Table 4.1 Wheat flour quality evaluation of the 17 pure wheat cultivars [a]

Cultivar	Wheat class[b]	Ash	Damaged starch	Zeleny	Hagberg	Protein	Wet gluten	HMW glutenin subunits			*Glu-1* score	Baking volume
		(%dm)	(%)	(mL)	(s)	(%dm)	(%)	***Glu-A1***	***Glu-B1***	***Glu-D1***		(cm^3/100 g flour)
Akteur	E	0.90 ± 0.01	4.75 ± 0.10	57 ± 1.5	441 ± 16	16.9 ± 0.1	37.2 ± 0.1	1	7+9	5+10	9	591 ± 12
Altos	E	0.77 ± 0.01	6.04 ± 0.02	55 ± 1.0	415 ± 4	13.3 ± 0.1	23.6 ± 1.2	null	17+18	5+10	8	530 ± 11
Anthus	A	0.79 ± 0.01	5.81 ± 0.09	41 ± 0.6	435 ± 9	13.7 ± 0.1	25.0 ± 0.6	null	7+9	2+12	5	541 ± 7
Astuce	B	0.56 ± 0.01	3.61 ± 0.15	37 ± 0.0	374 ± 7	11.6 ± 0.3	20.7 ± 0.5	1	17+18	5+10	10	429 ± 7
Bussard	E	0.60 ± 0.01	5.75 ± 0.04	70 ± 0.6	331 ± 7	15.8 ± 0.1	34.4 ± 0.6	1	7+9	5+10	9	632 ± 18
Constance	C	0.68 ± 0.00	4.16 ± 0.11	34 ± 0.6	370 ± 16	12.7 ± 0.2	24.1 ± 0.5	1	17+18	5+10	10	450 ± 6
Dinosor	A	0.68 ± 0.02	5.38 ± 0.14	51 ± 0.6	430 ± 10	13.3 ± 0.2	23.1 ± 0.7	1	6+8	5+10	8	455 ± 6
Enorm	E	0.73 ± 0.01	5.72 ± 0.03	64 ± 0.3	413 ± 11	14.6 ± 0.3	27.8 ± 0.5	1	7	5+10	8	584 ± 15
Ephoros	A	0.64 ± 0.01	5.43 ± 0.17	43 ± 0.0	347 ± 9	13.0 ± 0.2	24.8 ± 0.5	null	7+9	5+10	7	531 ± 8
Kodex	B	0.80 ± 0.01	5.96 ± 0.15	50 ± 0.0	487 ± 2	14.4 ± 0.1	29.3 ± 0.3	null	7+9	5+10	7	533 ± 13
Melkior	B	0.62 ± 0.01	6.32 ± 0.14	48 ± 0.6	382 ± 10	12.1 ± 0.2	27.3 ± 0.4	1	7	5+10	8	459 ± 7
Quebon	A	0.78 ± 0.01	5.99 ± 0.10	56 ± 0.6	487 ± 7	14.1 ± 0.1	29.7 ± 0.5	1	7+9	5+10	7	641 ± 5
Rosario	C	0.61 ± 0.01	5.38 ± 0.04	48 ± 0.0	418 ± 4	13.4 ± 0.1	25.2 ± 0.2	2*	17+18	2+12	8	536 ± 6
Rustic	B	0.78 ± 0.01	5.87 ± 0.12	55 ± 1.2	475 ± 20	14.7 ± 0.2	31.3 ± 0.2	null	6+8	5+10	6	506 ± 12
Samurai	B	0.60 ± 0.01	4.12 ± 0.09	39 ± 0.0	371 ± 4	12.3 ± 0.1	28.5 ± 0.3	2*	7+9	2+12	5	483 ± 8
Sokrates	A	0.63 ± 0.02	5.50 ± 0.05	50 ± 0.0	379 ± 4	14.1 ± 0.1	31.4 ± 0.3	1	6+8	5+10	8	557 ± 9
Tuareg	A	0.67 ± 0.02	5.46 ± 0.10	43 ± 0.0	401 ± 15	12.0 ± 0.2	23.8 ± 0.5	2*	7+9	2+12	7	508 ± 2
Min.		*0.56*	*3.61*	*34*	*331*	*11.6*	*20.7*				*5*	*429*
Max.		*0.90*	*6.32*	*70*	*487*	*16.9*	*37.2*				*10*	*641*

[a] mean and standard deviation of at least 3 repetitions
[b] according to the German wheat classification: E=elite (best quality); A=good quality; B=lower quality; c=feed wheat

The wheat cultivars used in this study are spread out over the four wheat classes (E, A, B and C) and this is also reflected in the breadmaking tests which pointed out large differences in bread volume (Table 4.1). Cultivars belonging to wheat class B or C resulted in the lowest bread volumes, cultivars Bussard (E) and Quebon (A) showed the highest volumes. Although a large variation in bread volume is seen, the majority of the wheat cultivars contains the HMW-GS pair 5+10 which can be associated with good breadmaking quality (Payne et al., 1981). Khan et al. (1989), on the other hand, stated that the presence of favorable combinations of HMW-GS does not guarantee good quality which is also confirmed in this study as cultivars Astuce and Constance, which have the highest *Glu-1* score, show very low bread volumes. In some cases it is possible that desired HMW-GS are being over expressed resulting in an over strong variety resulting in a lower baking performance.

4.5.2. Empirical rheology

4.5.2.1. Farinograph and alveograph results

The 17 wheat flours were analyzed for their empirical rheological properties with the farinograph and alveograph according to the standard protocols. The farinograph and alveograph properties are summarized in Annex 1. To determine the relevance to wheat quality control of the obtained parameters from the farinograph or alveograph, their correlations were further studied in more detail.

Among the farinograph parameters, stability, softening and quality number are highly correlated. Softening shows an inverse relationship with the stability ($r=-0.953^{**}$) and the quality number ($r=-0.647^{**}$). The quality number is mainly determined by the stability of the dough ($r=0.749^{**}$).

For the alveograph properties, an inverse relationship between the tenacity P and the extensibility L is observed ($r=-0.692^{**}$). The curve configuration ratio P/L is correlated with P ($r=0.950^{**}$), L ($r=-0.831^{**}$) and the deformation energy W ($r=0.565^{*}$). W is also related to P ($r=0.774^{**}$) and the elasticity index I_e ($r=0.681^{**}$).

Several of the obtained parameters are highly correlated which means that they merely contain similar information.

To get an overview of the variability of the dataset, PCA was performed on the farinograph and alveograph results for the 17 wheat cultivars (Figure 4.1).

For the farinograph results, the 1st and 2nd principal component explain 45.1 and 28.7% of the total variance, respectively. Figure 4.1A shows the score and loading plot for the first two principal components. The score plot shows the value of the principal components for the seventeen wheat cultivars. The loading plot illustrates how the original variables determine the principal components. For the farinograph properties, the first principal component is mainly determined by the dough stability (STAB) and the degree of softening (SOFT) during mixing. The wheat flours vary clearly in stability. The second principal component is mainly influenced by the water absorption (WA) and the dough development time (DDT). Most cultivars show high scores for the second principal component. Only cultivar Astuce is clearly separated from the other wheat samples which can be attributed to the low WA and DDT. WA, DDT and STAB are useful parameters for the evaluation of the flour strength. In general, the higher the value of these parameters, the stronger the flour (Rasper 1976).

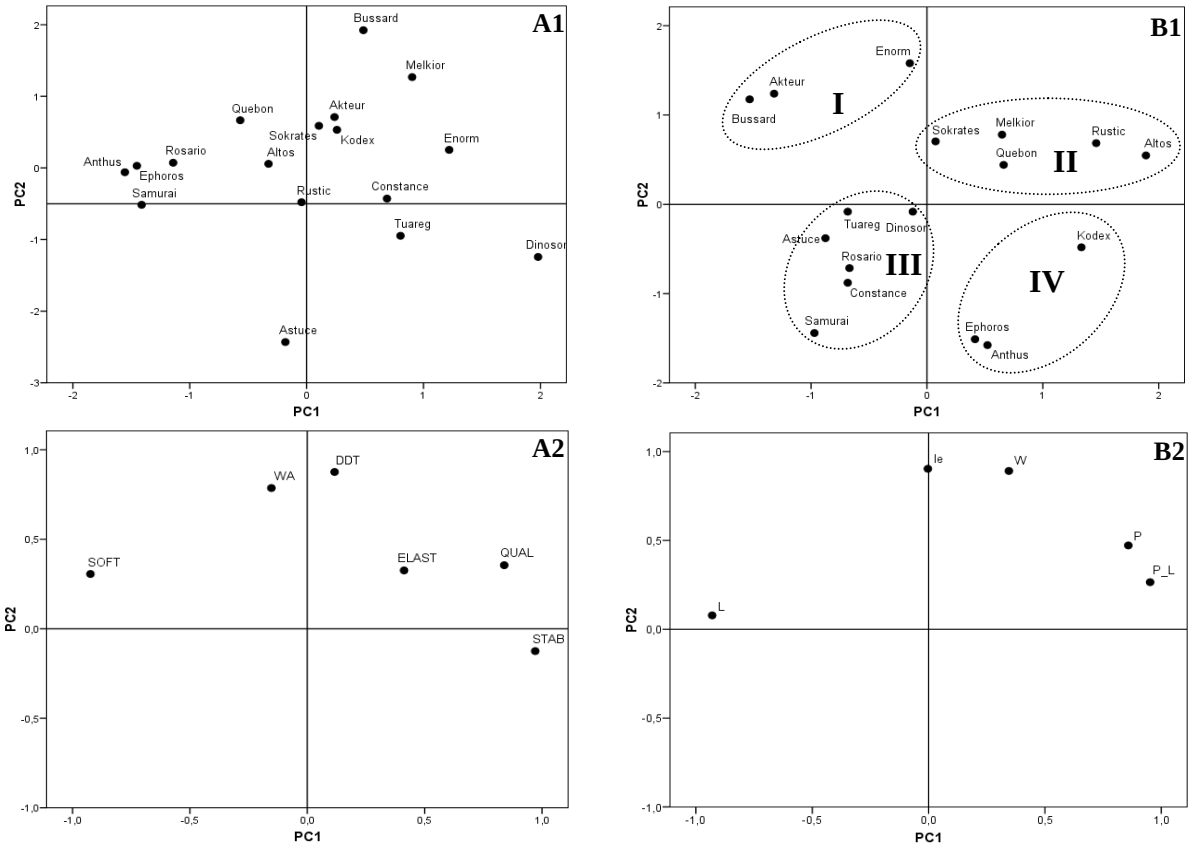

Figure 4.1 Score (1) and loading plots (2) of the first two principal components based on the farinograph (A) and alveograph (B) properties of 17 wheat cultivars

The same analysis was performed for the alveograph properties. The 1st and 2nd principal component explain 52.5 and 38.2% of the total variance, respectively. Figure 4.1B shows the score and loading plot for the first two principal components. The first principal component is mainly determined by the tenacity P and the extensibility L and the second principal component mainly by the deformation energy W and the elasticity index I_e. Based on the principal components, the wheat cultivars can be divided into four groups: [I] high W and high L; [II] high W and high P; [III] low W and high L; [IV] low W and high P. Wheat cultivars showing the lowest bread volumes are mainly present in group III. When comparing this with the wheat class classification, it is seen that no link exists between wheat class and alveograph properties except for group I, which consists of wheat cultivars only belonging to wheat class E (best baking quality).

It can be concluded that the dataset contains flour samples with a wide variety of empirical rheological properties.

4.5.2.2. Relationship between empirical rheology and flour quality

To investigate how flour properties such as protein content and damaged starch affect dough rheology as determined by the farinograph and alveograph, their relations were studied in more detail. Table 4.2 gives an overview of correlations found between selected empirical rheological properties and the flour quality parameters discussed in 4.5.1 for the 17 wheat flour cultivars.

Farinograph water absorption shows high correlations with damaged starch content, protein and wet gluten content, and Zeleny sedimentation value. When applying multiple linear regression with forward selection, it is found that 80% of the variance in farinograph water absorption can be explained by combination of damaged starch and protein content. This corresponds with the results from Tipples et al. (1978) who stated that farinograph water absorption can be predicted based on the amount of damaged starch content and the protein content. For durum wheat flour, starch damage was the predominant factor influencing the farinograph water absorption and protein content only exerted a moderate influence (Dexter et al., 1994). Also non-starch polysaccharides and more precisely arabinoxylans have been shown to influence the water absorption levels of a wheat flour (Roels et al., 1993). However, this was not part of this study.

Table 4.2 Pearson correlations (r) amongst empirical rheological properties and selected flour quality parameters[a]

	Protein	*Zeleny*	*Wet gluten*	*Damaged Starch*
WA	0.603*	0.758**	0.693**	0.811**
W	0.584*	0.888**	0.665**	0.687**
P	n.s.	0.530*	n.s.	0.805**
P/L	n.s.	n.s.	n.s.	0.675**

[a] significant at P<0.05 (*) and P<0.01 (**), n.s. = non significant

Alveograph P and W are well correlated with the farinograph water absorption (r=0.73**) and the amount of damaged starch. Increasing starch damage leads to higher and shorter alveograph curves (Dexter et al, 1994). W is also highly correlated with the Zeleny sedimentation value (r=0.888**). Farinograph elasticity correlates with L (r=0.746**) and I_e is related to the degree of softening (r=-0.660**) and the stability of the dough (r=0.718**).

Concerning the mixing requirements of the wheat flours, it was observed that dough development time (DDT) and dough stability (STAB) as determined in the farinograph were not significantly affected by protein content. However, DDT showed a positive correlation with wet gluten content (0.726**). No correlation was found between wet gluten and stability. It was also observed that dough stability was significantly influenced by the HMW-GS composition. STAB shows a positive correlation (r=0.503*) and SOFT a negative correlation (r=-0.500*) with the Glu-1 score. It thus seems that flours containing more gluten, need a longer mixing time for reaching full development and that the type of HMW-GS influences the stability of the dough during mixing and overmixing.

From these results, the complex interplay between protein content and protein quality, the amount of other flour constituents, water absorption and rheology is already apparent.

4.5.3. Rotational rheometry

4.5.3.1. Sample relaxation

The rheological properties of the flour-water doughs prepared from the 17 wheat flours, were analyzed by rotational rheometry. Their behaviour during sample relaxation after sample

compression, already indicated differences in rheological properties. Figure 4.2 shows the evolution of the NF after sample loading for three wheat cultivars (Bussard, Tuareg and Astuce). The dough of cultivar Astuce showed the highest NF (3 N) at equilibrium, dough of cultivar Bussard showed the lowest NF (0.5 N). All the other cultivars were situated in between.

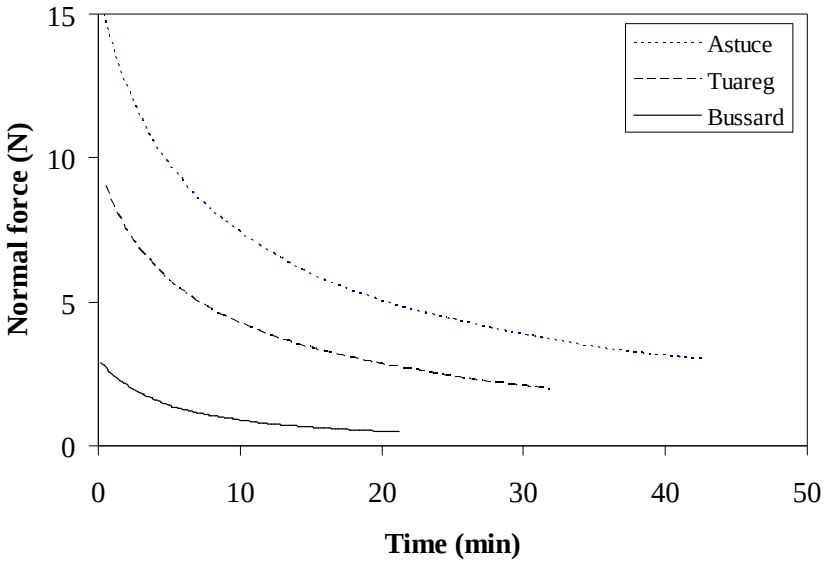

Figure 4.2. Evolution of the normal force as function of time for flour-water dough of three wheat cultivars (Bussard, Tuareg and Astuce) after sample loading

4.5.3.2. Dynamic oscillation

After sample loading, a time sweep experiment (0.01% strain - 1 Hz) was executed. An example of a time sweep experiment is shown in Figure 4.3. It is shown how the dynamic moduli (G', G" and $|G^*|$) decrease and the phase angle increases to reach a constant value. At the end of the time sweep the normal force (not shown in graph) also reached a constant value indicating adequate sample relaxation. The results for the dynamic moduli and phase angle δ are derived from the last 5 datapoints of the time sweep and thus reflect the small deformation viscoelastic properties of the relaxed dough sample, measured at a frequency of 1 Hz.

The results from the dynamic oscillation time sweep experiments for flour-water doughs prepared from the 17 different wheat flours are summarized in Annex 2. The coefficients of variation were lower than 5% for the dynamic moduli and lower than 1% for the phase angle δ. It can be observed that the elastic modulus G' has a higher value than the viscous modulus

G" for all dough samples. At low strains, a wheat flour dough behaves like an elastoviscous solid-like body (Weipert, 1990). Differences can be observed for the dynamic moduli among the wheat samples. G' varies between 6734 and 24502 Pa, G" between 2938 and 7974 Pa and phase angle δ between 18.0 and 24.6° This contrasts with previous reports in which no differences were observed in elastic modulus G' for bread doughs (Hayman et al., 1998; Safari-Ardi and Phan-Thien, 1998; Sliwinski et al., 2004a). It is found that the dynamic moduli G', G" and $|G^*|$ are strongly correlated ($r>0.99^{**}$). Phase angle δ shows inverse correlations with G' ($r=-0.908^{**}$), G" ($r=-0.887^{**}$) and $|G^*|$ ($r=-0.905^{**}$). In stead of G' or G", phase angle δ better reflects the viscoelastic status of a dough system.

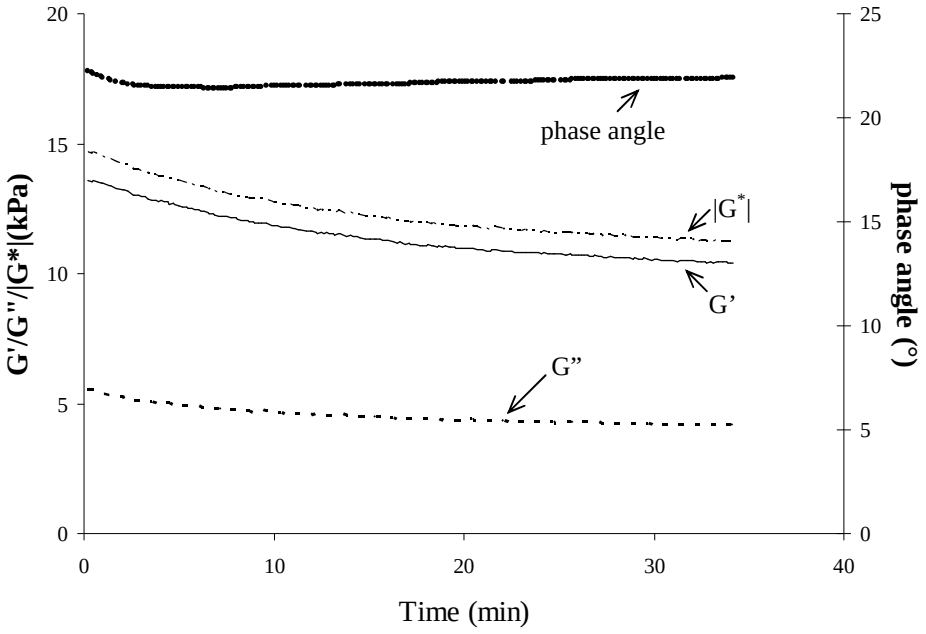

Figure 4.3 Time sweep experiment (0.01% - 1Hz) performed on the flour-water dough of wheat cultivar Quebon. Evolution of elastic modulus (G'), viscous modulus (G"), complex modulus ($|G^*|$) and phase angle is presented as function of time.

4.5.3.3. Creep-recovery

The time sweep measurements were followed by a creep-recovery test at a shear stress of 100 or 250 Pa. To illustrate, the creep-recovery curves of wheat varieties Bussard and Anthus are shown in Figure 4.4. The application of a higher shear stress, results in a higher creep deformation. The results of the creep-recovery measurements ($J_{c,max}$, $J_{r,max}$ and %recovery) for the 17 cultivars are reported in Annex 2. The coefficients of variation were lower than 6%.

The 17 wheat flour samples show a wide range of rheological behaviour, similar to what was observed from the farinograph and alveograph results.

When investigating the correlations between creep-recovery variables, it is found that max. creep compliance ($J_{c,max}$) and max. recovery compliance ($J_{r,max}$) are well correlated and this for both shear stresses of 100 Pa (r=0.991**) and 250 Pa (r=0.946**). But $J_{c,max}$ and $J_{r,max}$ show an inverse relationship with the relative recovery (% recovery) both at 100 Pa (r=-0.813** and -0.758**) and 250 Pa (r=-0.920** and -0.846**). These results indicate that when, at a constant shear stress, a higher creep deformation is induced, this results in a higher recovery but in a lower relative recovery (% of the creep deformation which is recovered) meaning that dough behaviour is more viscous. For most dough samples, $J_{r,max}$ showed a slight increase when applying a shear stress of 250Pa.

The results at 100 and 250 Pa are well correlated for $J_{c,max}$ (r=0.989**), $J_{r,max}$ (r=0.964**) and %recovery (r=0.928**). Tronsmo et al. (2003b) also found similar results between creep tests performed at 50 and 250 Pa for isolated gluten samples.

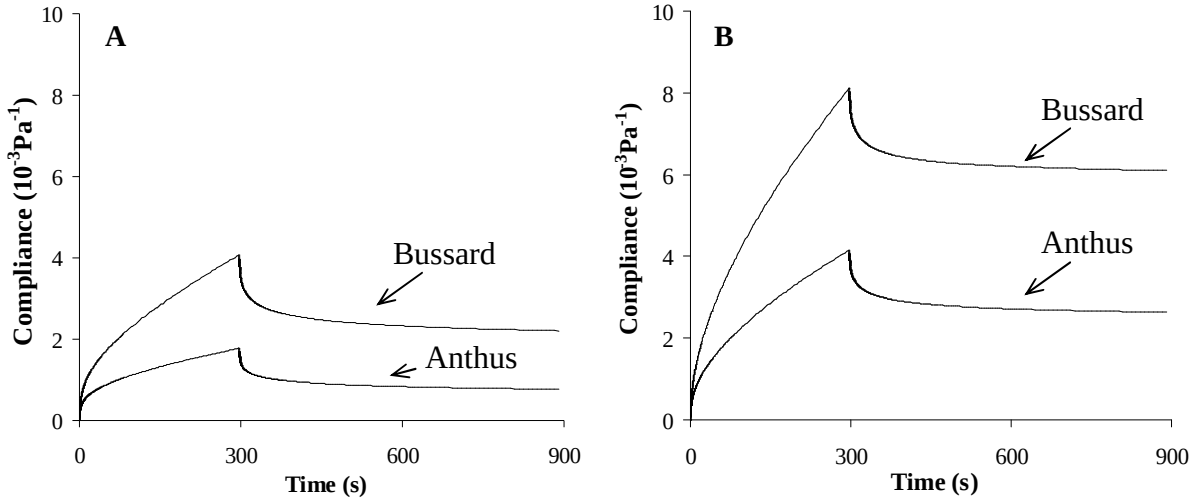

Figure 4.4 Example of creep-recovery curves for two wheat cultivars (Bussard and Anthus) with creep recorded under a shear stress of 100 Pa (A) and 250 Pa (B)

4.5.3.4. Relation between dynamic oscillation and creep-recovery parameters

Dynamic oscillation and creep-recovery parameters were obtained from the same dough systems. Although both methods are performed in shear, dynamic oscillation measurements were performed in the LVR and creep-recovery measurements, in this case, caused

deformations beyond the LVR. Therefore it is interesting to compare the obtained results and relate deformations occurring outside the LVR with small deformation rheological properties inside the LVR reflecting dough microstructure.

A non-linear relationship between the dynamic moduli and $J_{c,max}$ was found and this for both shear stresses ($r^2>0.9$). In Figure 4.5A-B an example is shown of this relation for the complex modulus $|G^*|$ and $J_{c,max}$ obtained at 100 and 250 Pa. As $J_{c,max}$ and $J_{r,max}$ are strongly correlated ($r>0.9^{**}$), similar results were found with $J_{r,max}$.
It can be seen that $J_{c,max}$ stays relatively stable for $|G^*| > 15000$ Pa. $J_{c,max}$ increases steadily between 15000 and 10000 Pa and finally shows a sharp rise when $|G^*|$ falls below 10000 Pa. As the consistency of the dough system ($|G^*|$) decreases, the deformation occurring when larger shear stresses are involved increases according to a power law relationship.
When relating $J_{c,max}$ to the phase angle δ, also a power law relation was found (Figure 4.5C-D). This is again an indication of the more than proportional increase of creep deformation when a dough system behaves less elastic at small deformation.

On the other hand, a linear relationship can be observed between the phase angle δ and $J_{r,max}$ ($r\geq0.9^{**}$) at both shear stresses (Figure 4.5E-F). In contrast to the creep deformation, $J_{r,max}$ does linearly increase with increasing δ. This means that the increase in creep deformation is attributed to the viscous component in the dough system. It is also observed that $J_{r,max}$ remains within the same range for both shear stresses. This corresponds to the plateau which was found for $J_{r,max}$ when increasing the shear stress from 10 to 1000 Pa (3.4.2.4). $J_{r,max}$ remains constant or increases slightly when the shear stress is increased from 100 to 250 Pa.

These results indicate that the information obtained from small deformation rheological measurements of wheat flour dough can be related to large deformation rheological properties. The small deformation viscoelastic properties may indicate how the dough system will behave under the application of a large shear deformation, for example during dough processing.

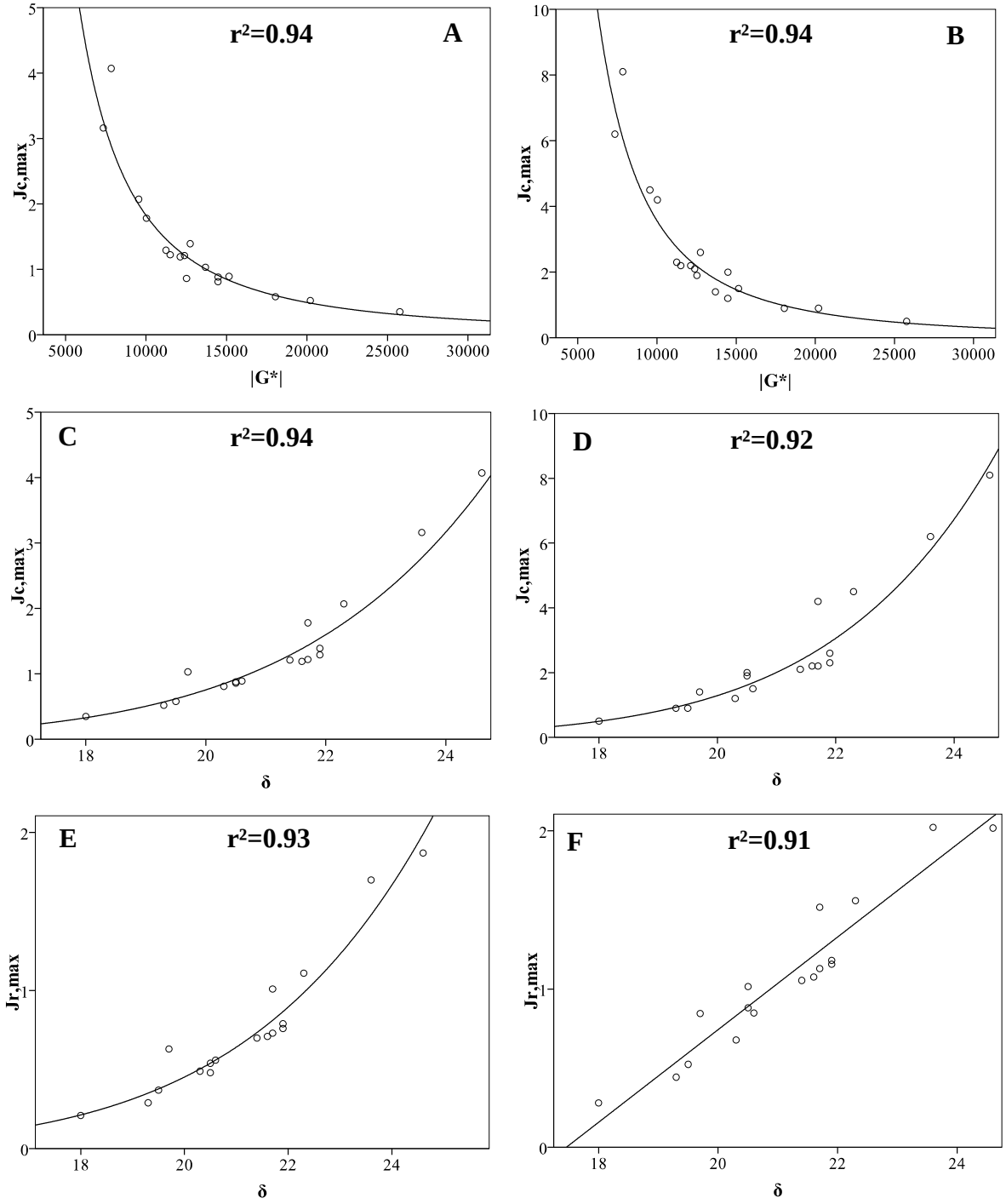

Figure 4.5 Relationships between rheological parameters obtained from dynamic oscillation and creep-recovery. [A] $J_{c,max}$ (100Pa) and |G*|; [B] $J_{c,max}$ (250Pa) and |G*|; [C] $J_{c,max}$ (100Pa) and δ; [D] $J_{c,max}$ (250Pa) and δ; [E] $J_{r,max}$ (100Pa) and δ; [F] $J_{r,max}$ (100Pa) and δ. $J_{c,max}$/ $J_{r,max}$ and δ are expressed in units [10^{-3} Pa^{-1}] and [°] respectively. Datapoints represent the 17 investigated wheat cultivars (Annex 2). Regression coefficients of the best fitting curve are indicated in the graph.

4.5.4. Relationship between rotational rheometry, standard flour quality parameters and empirical rheology

To get more insight in the factors which control the rheological properties as determined by rotational rheometry, the relationship between the obtained parameters and standard flour quality descriptives (4.5.1) was investigated. Also, rheological parameters from rotational rheometry were compared to the rheological parameters obtained form the farinograph and the alveograph (4.5.2.1), which are used in standard quality control of wheat flour.

4.5.4.1. Dynamic oscillation

Table 4.3 summarizes the most important correlations between dynamic oscillation, standard flour quality parameters and empirical rheology. Dynamic oscillation parameters are highly influenced by dough water content and protein content of the wheat flour as they show significant but negative correlations with protein content ($r=-0.7^{**}$), Zeleny sedimentation value ($r=-0.6^{**}$), damaged starch content ($r=-0.5^{*}$) and farinograph water absorption ($r=-0.7^{**}$). The dynamic moduli show an inverse relationship and phase angle δ a positive correlation with water absorption. No relationship was found between farinograph or alveograph parameters and the dynamic rheological properties, except for water absorption.

Table 4.3 Pearson correlations (r) amongst dynamic moduli and phase angle δ and selected quality parameters[a]

	Protein	*Zeleny*	*Wet gluten*	*Damaged Starch*	*WA*	*L*
G'	-0.736^{**}	-0.640^{**}	-0.669^{**}	-0.538^{*}	-0.744^{**}	n.s.
G"	-0.735^{**}	-0.621^{**}	-0.669^{**}	-0.515^{*}	-0.714^{**}	n.s.
$\|G^{*}\|$	-0.735^{**}	-0.635^{**}	-0.666^{**}	-0.536^{*}	-0.740^{**}	n.s.
Phase angle δ	0.802^{**}	0.710^{**}	0.691^{**}	n.s.	0.731^{**}	n.s.

[a] significant at $P<0.05$ (*) and $P<0.01$ (**), n.s. = non significant

It has been reported that protein content and water content of the dough influence the dynamic rheological parameters. Protein content and water absorption are closely linked (Bloksma, 1990a). Water is an important factor in determining the viscoelastic properties of dough. It has a dual role because it can act as an inert filler reducing the dynamic properties proportionally and as a lubricant enhancing the relaxation (Masi et al., 1998). Several authors

reported a decrease of the elastic and viscous modulus with increasing water content (Hibberd and Wallace, 1966; Letang et al., 1999; Masi et al., 1998; Navickis et al., 1982). Some authors also observed that an increase of the water content did not affect tan δ (Hibberd, 1970a; Letang et al., 1999). However, Masi et al. (1998) only observed this in the high frequency range (10-100Hz). A decrease of the dynamic moduli was reported for doughs with increasing gluten content and prepared with the optimal water content (Hibberd, 1970b; Smith et al., 1970; Uthayakumaran et al., 2002).

Figure 4.6 illustrates the dependency of |G*| on water absorption and protein content. When looking at the effect of water absorption in more detail, it can be observed that for the flours with the lowest water absorption (±50%), indeed higher |G*| have been measured. For the water absorption range from 53 to 63% the effect on |G*| is less clear. For example, several samples have a water absorption of ±59%, but differ largely in the measured |G*|. Furthermore, samples with similar |G*|, differ largely in their water absorption.

When looking at the effect of protein content, similar observation can be made. The samples showing the highest (±16%) and lowest (±11%) protein contents, result in the lowest and highest measured |G*| for their flour-water dough system. On the other hand, several samples with similar protein content, show significant differences in |G*| and similar |G*| values are found for samples differing largely in protein.

So, it can be concluded that extremes in water absorption and protein content, indeed largely influence the measured small deformation properties of a dough system. But in between the extremes, it is thought that the small deformation properties are reflecting the structural organisation of the dough system as a result of the effective water distribution in the dough and the interactions existing between the different dough components.

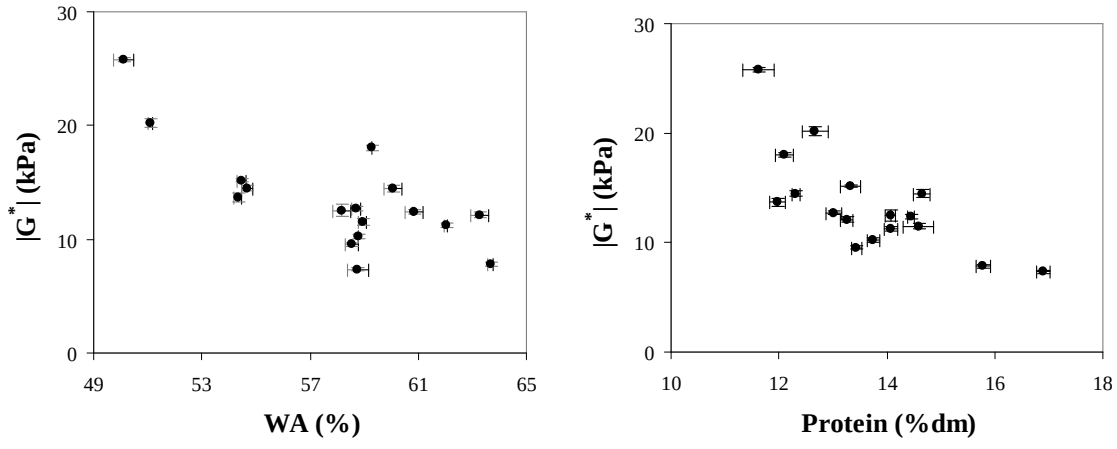

Figure 4.6 Dependency of |G*| on farinograph water absorption (WA) and flour protein content

4.5.4.2. Creep-recovery

The same correlation analysis was performed for the creep-recovery parameters. Compared with dynamic oscillation, creep-recovery parameters are less sensitive for dough water content, but are also largely influenced by protein content (Table 4.4). The maximum deformation obtained in the creep-recovery test is related to the protein content ($r>0.7^{**}$), Zeleny sedimentation value ($r\geq0.6^{**}$) and farinograph water absorption ($r=0.5^{*}$-0.6^{**}). A higher protein content and farinograph water absorption seem to lead to higher creep and recovery compliances. However, the influence of water absorption is much smaller compared to dynamic oscillation. Creep-recovery behaviour is mainly determined by the protein content of the dough system. As a dough system contains more protein and water, the relative amount of the continuous gluten phase will increase in expense of the starch phase. This will favour deformation under creep-recovery.

Wang and Sun (2002) also reported positive correlations between $J_{r,max}$, protein content and farinograph water absorption.

Table 4.4 Pearson correlations (r) amongst fundamental rheological properties and selected flour quality parameters[a]

	Protein	*Zeleny*	*Wet gluten*	*Damaged Starch*	*WA*	*L*
Creep-recovery 100 Pa						
$J_{c,max}$	0.746^{**}	0.671^{**}	0.644^{**}	n.s.	0.533^{*}	0.627^{**}
$J_{r,max}$	0.775^{**}	0.622^{**}	0.657^{**}	n.s.	0.559^{*}	0.575^{*}
% Recovery	n.s.	n.s.	n.s.	n.s.	n.s.	-0.722^{**}
Creep-recovery 250 Pa						
$J_{c,max}$	0.720^{**}	0.557^{*}	0.619^{*}	n.s.	0.512^{*}	0.632^{**}
$J_{r,max}$	0.767^{**}	0.587^{*}	0.644^{**}	n.s.	0.607^{**}	n.s.
% Recovery	-0.577^{*}	n.s.	n.s.	n.s.	n.s.	-0.628^{**}

[a] significant at $P<0.05$ (*) and $P<0.01$ (**), n.s. = non significant

For the creep-recovery parameters, a correlation with alveograph extensibility was found. A flour which gives a more extensible dough in the alveograph test, will be likely to show a higher deformation in creep-recovery experiments. A higher value for L is also an indication for a lower % recovery in a creep test. For durum wheat flours it was found that $J_{r,max}$ showed a strong inverse relationship with alveograph P/L and W (Edwards et al., 1999). It would be interesting to investigate if correlations could be improved by testing similar dough formulations on the alveograph as by creep-recovery.

As mentioned before, a large span in protein content is present among the wheat flours. Correlation analysis has shown that protein content has a major effect on dough rheological properties as measured by rotational rheometry. If other samples were collected with little variation in protein content and large differences in gluten protein strength, a different impact could be expected on the various rheological properties.

4.5.5. Flour and dough properties in relation to bread volume

4.5.5.1. Pearson correlations

To investigate the relationship between bread volume and the studied flour and dough characteristics, Pearson correlations were determined. Table 4.5 gives an overview of the significant correlations between the flour and dough properties and bread volume. For standard flour quality parameters, correlations with bread volume are found for protein content ($r=0.750^{**}$), farinograph water absorption ($r=0.748^{**}$) and Zeleny sedimentation value ($r=0.739^{**}$). Figure 4.7A illustrates the relation between bread volume and WA. Figure 4.7B illustrates the relation between bread volume and protein content. Similar correlations between bread volume and protein content or WA have been reported (Dobraszczyk and Salmanowicz, 2008; Konopka et al., 2004; Park et al., 2006). From the alveograph parameters, W shows a rather low correlation with bread volume ($r=0.552^{*}$). No other parameter obtained from empirical rheology showed a relation with bread volume.

Table 4.5 Significant Pearson correlations (r, significant at P<0.05 (*) and P<0.01 (**)) between flour and dough properties and bread volume

Variable	*r*
Phase angle δ	0.833^{**}
G'	-0.827^{**}
G"	-0.824^{**}
$\|G^*\|$	-0.824^{**}
Protein	0.750^{**}
Zeleny	0.739^{**}
Wet gluten	0.709^{**}
WA	0.748^{**}
W	0.552^{*}
$J_{c,max}$ (100 Pa)	0.706^{**}
$J_{r,max}$ (100 Pa)	0.736^{**}
$J_{c,max}$ (250 Pa)	0.689^{*}
$J_{r,max}$ (250 Pa)	0.790^{**}
% recovery (250 Pa)	-0.548^{*}

Of all the studied parameters, phase angle δ (Figure 4.7D) and the dynamic moduli show the highest correlations with the bread volume. The dynamic moduli show a significant linear correlation with the bread volume. However, their relationship is best described by a power law equation. A power law relationship can be established between the bread volume and G' (r^2=0.75), G" (r^2=0.72) and the complex modulus $|G^*|$ (r^2=0.74) which is illustrated in Figure 4.7C. These results contrast with other authors which were not able to relate dynamic rheological properties of wheat flour dough and baking volume (Janssen et al., 1996a; Kokelaar et al., 1996; Autio et al., 2001; Khatkar and Schofield, 2002). Khatkar and Schofield (2002) mentioned an inverse relationship between G' and the loaf volume for 12 wheat cultivars of diverse breadmaking performance, but this relation was very weak (r^2=0.16). Autio et al. (2001) only achieved a good correlation (r=0.72) with the baking performance if all data points of G' in a stress curve were evaluated with multivariate analysis.

For the creep-recovery parameters, $J_{r,max}$ at 250 and 100 Pa show the highest correlation with the bread volume. Figure 4.7E illustrates the relation between bread volume and $J_{r,max}$. These results correspond with those of Wang and Sun (2002) who found a high correlation (r>0.9) between the maximum recovery strain and the bread volume. Also $J_{c,max}$ shows a relatively high correlation with bread volume, in contrast to the results of Wang and Sun (2002) where maximum creep strain badly correlated with loaf volume. These contrasting results may be explained by the different test setup used (compression instead of shear) which possibly has an impact on the creep deformation behaviour.

These results suggest that when the overall consistency of the flour-water dough decreases (higher phase angle, lower moduli, higher creep and recovery deformation), the bread formulation dough will show a larger expansion ability in breadmaking (larger bread volumes). However, this does not give information on the ability of the dough membranes to strengthen the gas cell membranes and thus prevent premature rupture.

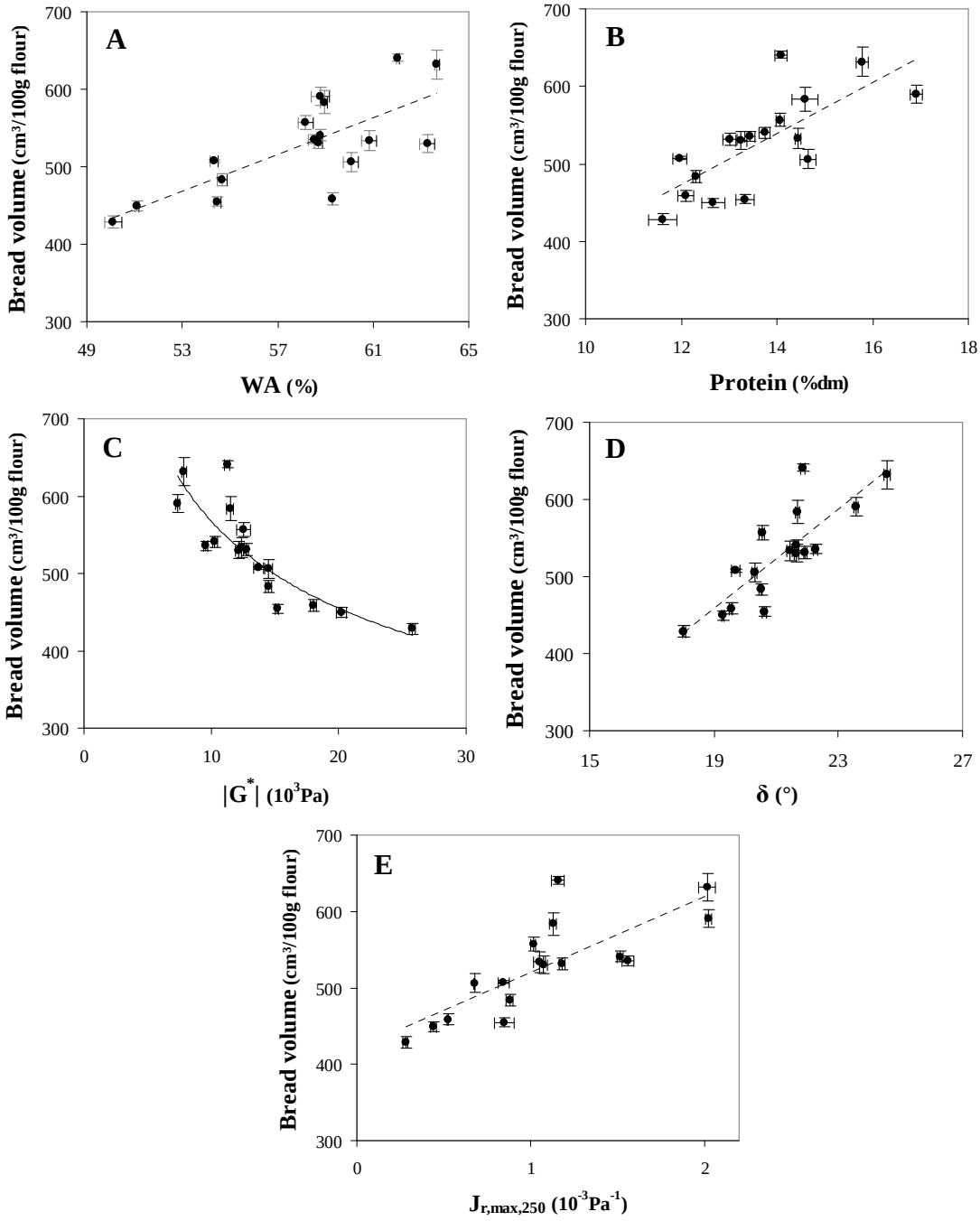

Figure 4.7 Relation between bread volume and selected parameters: [A] WA, [B] protein content, [C] $|G^*|$, [D] phase angle, [E] $J_{r,max,250}$

4.5.5.2. Multiple linear regression

In order to obtain a better prediction of the bread volume, multiple linear regression with forward model selection was used to predict the bread volume with one or more quality parameters. By applying multiple linear regression with forward model selection, on different groups of the obtained variables, only parameters which could significantly improve the

regression model, were added. The results of the multiple linear regression are summarized in Table 4.6. Besides r^2 also r^2_{adj} is given, which takes into account the number of variables used in the regression. Also, the standard error of the estimate is provided which is an indication of the error present on the predicted value.

From the standard flour properties, protein content (r^2=0.56, r^2_{adj}=0.53) and Zeleny sedimentation (r^2=0.55, r^2_{adj}=0.52) value can explain around half of the variance in bread volume. If only empirical rheological properties are considered, a combination of WA and the curve configuration ratio P/L can best predict bread volume (r^2=0.75, r^2_{adj}=0.72). When combining the flour properties with the empirical rheological properties, a combination of protein content, WA and P/L can explain 80% of the variance in bread volume (r^2=0.80, r^2_{adj}=0.75). If only two parameters are used, a combination of Zeleny sedimentation value and the dough stability (r^2=0.76, r^2_{adj}=0.73) gives the best prediction.

Table 4.6 Multiple linear regression with forward model selection and bread volume as dependent variable

Variables entered	r^2	r^2_{adj}	*St. error of the estimate*
Protein	0.56	0.53	42
Protein + WA	0.70	0.66	36
Protein + WA + P/L	0.80	0.75	31
Zeleny	0.55	0.52	43
Zeleny + STAB	0.77	0.74	32
Zeleny + SOFT	0.76	0.73	32
WA	0.56	0.53	42
WA + P/L	0.75	0.72	33
W	0.31	0.26	53
W + STAB	0.58	0.52	43
W + SOFT	0.60	0.54	42
W + STAB + ELAST	0.70	0.63	38
W + STAB + P/L	0.76	0.70	34
Phase angle δ	0.69	0.67	35
$J_{r,max}$ (250 Pa)	0.62	0.60	39
$J_{r,max}$ (250 Pa) + Zeleny	0.74	0.70	34
$J_{r,max}$ (250 Pa) + WA	0.74	0.70	34
$J_{r,max}$ (250 Pa) + W	0.74	0.71	33

It was not possible to combine another parameter with phase angle δ to improve the predictive power of the regression model. Maximum recovery compliance at 250 Pa can, on its own, explain 62% of the variance in bread volume (r^2=0.62, r^2_{adj}=0.60). When combined with Zeleny sedimentation value, WA or W, the explained variance increases to 74%. The plots of predicted versus measured bread volumes for four different regression models are shown in Figure 4.8.

It can be observed that the best prediction is given by combining protein content, WA and P/L. This model reflects the interaction between protein content, which is known to largely determine breadmaking potential of wheat flour (Finney and Barmore, 1948), water absorption, which is determined by flour composition, and the resulting viscoelastic balance of the dough system as shown in alveograph P/L. The model statistics are summarized in Annex 3. The sign of their respective coefficients in the model shows how the different parameters contribute to the model prediction. It is found that wheat flours having a higher protein content and water absorption will give a higher bread volume. However, P/L has a negative contribution to the model. Thus a lower P/L value, indicating higher dough extensibility and thus less dough strength, is related to higher bread volumes.

So, it can be concluded that the interaction between protein content, water absorption and dough extensibility largely determines the breadmaking potential of the wheat flours studied in this dataset. As discussed before, phase angle δ reflects structural organization of the dough system as a result of the effective water distribution in the dough and the interactions existing between the different dough components. This may explain why phase angle delta, on its own, shows a high correlation with the bread volume for this data set.

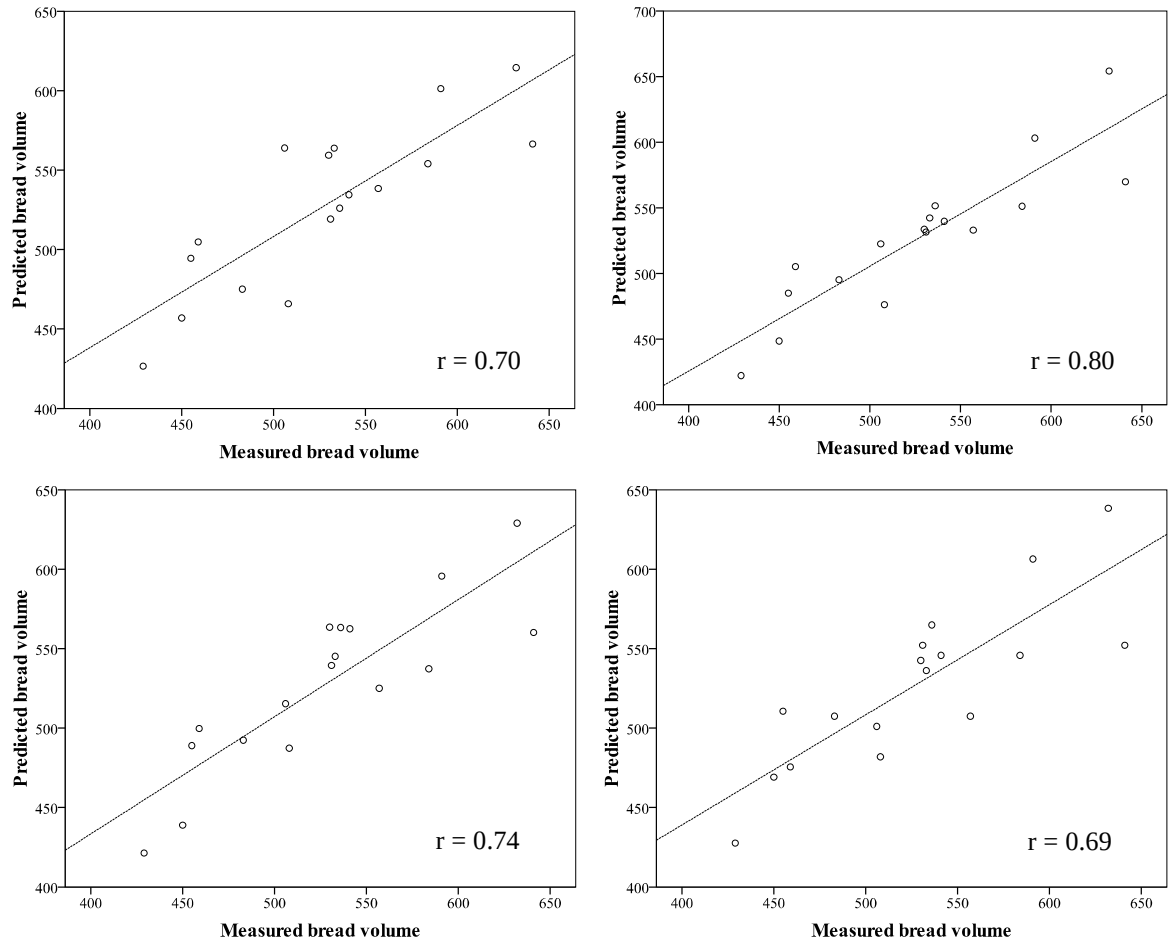

Figure 4.8 Predicted versus measured bread volumes for selected models obtained by multiple linear regression with forward selection. Variables entered: (A) protein and WA; (B) protein, WA and P/L; (C) $J_{r,max,250}$ and WA; (D) phase angle δ

4.5.5.3. Model validation

A selection of the predictive models listed in Table 4.6 was tested for two wheat cultivars (Cubus and Haussmann) which were not included in the original set of wheat cultivars. The selected quality parameters of these two cultivars are given in Table 4.7. The generated regression models were used to predict the bread volume of the two cultivars and the deviation to the measured bread volume is given in terms of percentage Table 4.8. Most regression models overestimate the bread volume, only model V results in an underestimation. In general, bread volume of Hausmann was better predicted than that of Cubus. The model that showed the best relation with bread volume as determined by multiple linear regression (model II: protein-WA-P/L), could also give the best prediction of the bread volume of Cubus and Hausmann. This model thus seems to offer the most potential for use in

quality control of wheat flour. The best prediction obtained from a model containing a parameter from rotational rheometry was that of model IX ($J_{r,max,250}$ and W).
Regression models may be improved by expanding the underlying dataset with more samples.

Table 4.7 Selected quality parameters for wheat cultivars Cubus and Haussmann[a]

Cultivar	Wheat class	Protein (%dm)	Zeleny (mL)	WA (%)	STAB (min)	W (10^{-4} J)	P/L (-)
Cubus	A	12.4 ± 0.1	59 ± 1.7	60.6 ± 0.2	8.7 ± 0.8	227 ± 1.0	1.79 ± 0.15
Haussmann	B	12.1 ± 0.1	43 ± 0.6	62.1 ± 0.1	4.4 ± 0.2	158 ± 3.5	1.07 ± 0.03

Cultivar	$\lvert G^* \rvert$	Phase angle	$J_{r,max,250}$ (%)	Baking volume (cm^3/100 g flour)	HMW glutenin subunits *Glu-A1*	*Glu-B1*	*Glu-D1*
Cubus	14642 ± 322	21.3 ± 0.1	0.90 ± 0.06	467 ± 5	null	7	5+10
Haussmann	10375 ± 149	21.6 ± 0.1	1.43 ± 0.05	522 ± 7	2*	7+9	2+12

[a] Mean and standard deviation based on three repetitions are presented

Table 4.8 Prediction of the bread volume for wheat cultivars Cubus and Haussmann by using regression models obtained from multiple linear regression analysis

n°	Variables entered	Cubus	Haussmann
Measured bread volume		467±5	522±7
Predicted bread volume and deviation to the measured value (%)			
I	Protein + WA	522 (12%)	525 (1%)
II	Protein + WA + P/L	482 (3%)	540 (4%)
III	Zeleny + STAB	603 (29%)	550 (5%)
IV	WA + P/L	490 (5%)	571 (10%)
V	W + STAB + P/L	454 (-3%)	481 (-8%)
VI	$J_{r,max,250}$	510 (9%)	563 (8%)
VII	$J_{r,max,250}$ + Zeleny	542 (16%)	535 (3%)
VIII	$J_{r,max,250}$ + WA	533 (14%)	578 (11%)
IX	$J_{r,max,250}$ + W	505 (8%)	526 (1%)
XI	Phase angle	532 (14%)	544 (4%)

4.5.6. Burgers model

To this point, only three parameters from the creep-recovery curves were used more precisely $J_{c,max}$, $J_{r,max}$ and %recovery. However, as has been shown in Chapter 3, the Burgers model can be used to describe the creep and recovery curves. Extra parameters are thus obtained under the form of instantaneous or retarded compliances and retardation times. The 6-parameter

Burgers model was applied to the creep-recovery curves obtained at shear stresses of 100 and 250 Pa. The results are summarized in Annex 4 for the creep curves and in Annex 5 for the recovery curves.

To get a better view on the results and to characterize samples with similar rheological properties, PCA was performed on the model parameters obtained from the creep-recovery curves at 100 Pa and 250 Pa separately. For the results obtained at 100 Pa, score and loading plot are shown in Figure 4.9. PC1 and PC2 explain 84.6 and 8.8 % of the total variance, respectively. Thus more than 90% of the variability present in the dataset, can be reduced to only two principal components. From the loading plot it can be seen that PC1 mainly is determined by instantaneous and retarded compliances, of both creep and recovery, and steady state viscosity. It seems that instantaneous and retarded compliances contain similar information as they are placed together on the loading plot. In 4.5.3.3 it was also concluded that maximum creep and recovery compliances were highly correlated, even between measurements made at different shear stress. PC2 mainly contains information from the retardation times, especially retardation time r_2 from the recovery curve. However, in the score plot no clear separation can be observed among the wheat samples as they are scattered rather regularly around the centre point of the score plot.

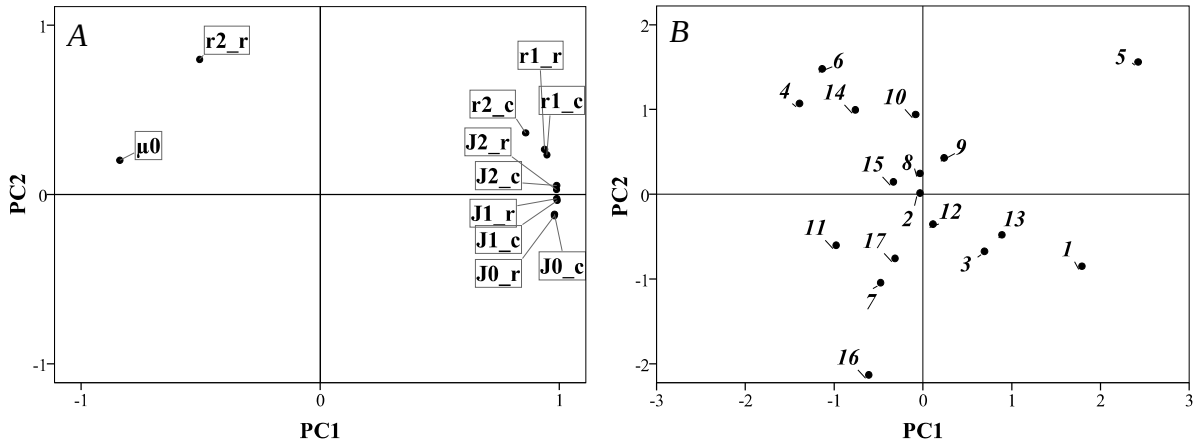

Figure 4.9 Loading (A) and score (B) plots of the first two principal components (PC) based on parameters obtained from modelling of the creep-recovery curves (100 Pa) of 17 wheat cultivars. Parameters shown in the loading are labeled with _c or _r if they are derived from creep or recovery data respectively. Numbers in the score plot correspond to the wheat cultivars as listed in Table 4.9.

A similar approach was applied to the results obtained from the creep-recovery curves at 250 Pa (Figure 4.10). PC1 and PC2 explain 80.6 and 10.8 % of the total variance, respectively. Again, most of the variability in the dataset can be reduced to two principal components. Information in PC1 is mainly determined by instantaneous and retarded compliances of creep and recovery, together with the creep retardation times and the steady state viscosity. Recovery retardation times, especially r_2, control the information in PC2. When looking at the score plot (Figure 4.10B), three groups of wheat cultivars can be observed with similar viscoelastic properties. Wheat cultivars in group I show low to medium deformability under shear (low compliances and high steady state viscosity) and lower values for r_2. Group II gathers samples with medium deformation properties and higher r_2. Cultivars from group III especially show a very large deformability and have r_2 values comparable to Group I.
Thus it seems that creep-recovery measurements performed at a sufficiently high shear stress are able to classify wheat samples according to similar viscoelastic properties.

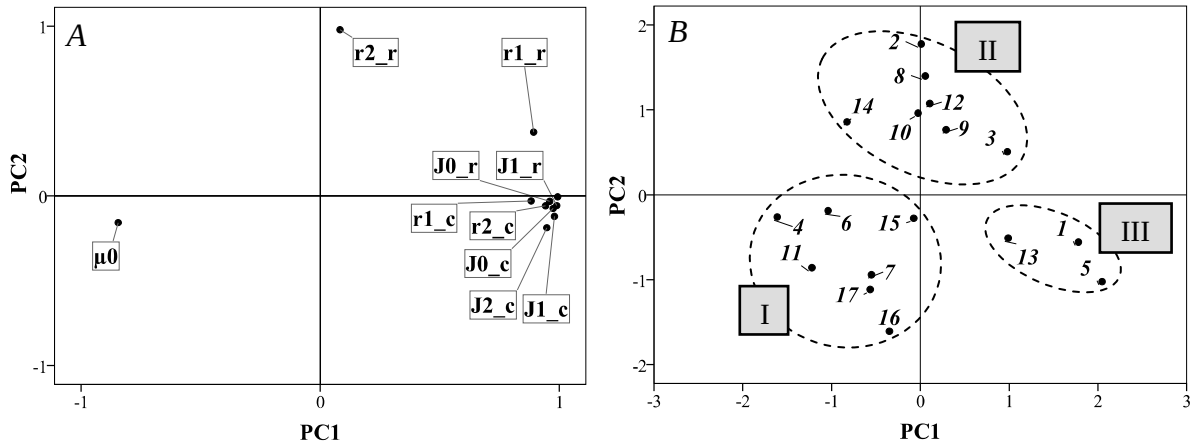

Figure 4.10 Loading (A) and score (B) plots of the first two principal components (PC) based on parameters obtained from modelling of the creep-recovery curves (250 Pa) of 17 wheat cultivars. Parameters shown in the loading are labeled with _c or _r if they are derived from creep or recovery data respectively. Numbers in the score plot correspond to the wheat cultivars as listed in Table 4.9.

In Table 4.9 the major quality characteristics of the three groups of wheat cultivars are listed. It is clear that the classification according to the viscoelastic properties deviates from the wheat class labels as wheat cultivars of low, good and excellent baking quality are spread out over the three groups. On the other hand, the classification reflects quite well the baking results as presented by baking volume. Wheat flour samples which resulted in the lowest baking volume are predominant in Group I. This corresponds to the lower values found for

protein content, Zeleny sedimentation value and farinograph water absorption (all below 60%). The flour-water doughs of these samples show a low deformation and high steady state viscosity under shear at high shear stress which seems to be linked to limited dough expansion during breadmaking. When deformed, they also show a fast elastic recovery.

On the other side, good to excellent bread volumes are found in both group II and III. Wheat samples in these groups generally have higher protein content, Zeleny sedimentation value and farinograph water absorption. This results in wheat samples with medium or standard (group II) and high (group III) deformability in creep. Although excellent baking volumes occur in both groups, the wheat cultivars differ in their rheological properties. This may be important as different behaviour during dough processing (e.g. sheeting) may be expected.

Table 4.9 Quality characteristics of the wheat cultivars divided in three groups according to the principal component analysis based on their viscoelastic properties*

Wheat cultivar		Wheat class**	Protein (%dm)	Zeleny (mL)	*Glu-1* score	WA (%)	Baking volume (cm^3/100 g flour)
Group I							
4	Astuce	B	11.6 ± 0.3	37 ± 0.0	10	50.1 ± 0.4	429 ± 7
6	Constance	C	12.7 ± 0.2	34 ± 0.6	10	51.1 ± 0.1	450 ± 6
7	Dinosor	A	13.3 ± 0.2	51 ± 0.6	8	54.5 ± 0.1	455 ± 6
11	Melkior	B	12.1 ± 0.2	48 ± 0.6	8	59.3 ± 0.0	459 ± 7
15	Samurai	B	12.3 ± 0.1	39 ± 0.0	5	54.7 ± 0.2	483 ± 8
16	Sokrates	A	14.1 ± 0.1	50 ± 0.0	8	58.2 ± 0.3	557 ± 9
17	Tuareg	A	12.0 ± 0.2	43 ± 0.0	7	54.3 ± 0.2	508 ± 2
Group II							
2	Altos	E	13.3 ± 0.1	55 ± 1.0	8	63.3 ± 0.3	530 ± 11
3	Anthus	A	13.7 ± 0.1	41 ± 0.6	5	58.8 ± 0.0	541 ± 7
8	Enorm	E	14.6 ± 0.3	64 ± 0.3	8	58.9 ± 0.2	584 ± 15
9	Ephoros	A	13.0 ± 0.2	43 ± 0.0	7	58.7 ± 0.2	531 ± 8
10	Kodex	B	14.4 ± 0.1	50 ± 0.0	7	60.8 ± 0.3	533 ± 13
12	Quebon	A	14.1 ± 0.1	56 ± 0.6	7	62.0 ± 0.1	641 ± 5
14	Rustic	B	14.7 ± 0.2	55 ± 1.2	6	60.1 ± 0.3	506 ± 12
Group III							
1	Acteur	E	16.9 ± 0.1	57 ± 1.5	9	58.8 ± 0.4	591 ± 12
5	Bussard	E	15.8 ± 0.1	70 ± 0.6	9	63.7 ± 0.1	632 ± 18
13	Rosario	C	13.4 ± 0.1	48 ± 0.0	8	58.5 ± 0.3	536 ± 6

* mean and standard deviation of at least 3 repetitions

** according to the German wheat classification: E=elite (best quality); A=good quality; B=lower quality; C=feed wheat

Although PC2 only explains 10.8% of the total variance of the results at 250 Pa, recovery retardation times r_1 and r_2 are crucial in grouping the wheat cultivars with similar rheological properties. Especially r_2 seems to be an interesting parameter which may contain information

regarding the viscoelastic properties that relate to baking quality. Tronsmo et al. (2003c) reported significantly lower retardation times for strong (HMW-GS 5+10) wheat cultivars compared to the weak (HMW-GS 2+12) cultivars. This indicates that stronger glutens show a faster recovery after creep. However, no relation with bread volume was reported. In this research, no relationship between retardation time of the flour-water doughs and HMW-GS composition was found. Also, no correlation between r_2 and bread volume could be established. On the other hand, when looking at Group I (low bread volumes) and Group II (good to excellent bread volumes), r_2 is a relatively good parameter for classifying the wheat cultivars according to their bread volume potential. It then seems that higher bread volumes are obtained from doughs which show a higher retardation time r_2 and thus demonstrate a slower elastic response after creep. In contrast, Kawai et al. (2006) found an inverse relationship of retardation time with bread volume. However, only three wheat cultivars at different water contents were studied.

The observation that wheat cultivars of Group III exhibited lower retardation times may be due to the large deformations obtained for those samples. It was already shown that recovery retardation times are very sensitive to conditions of the creep-recovery measurement as duration of the creep phase (3.4.2.3) and applied shear stress (3.4.2.4).

4.6. Conclusions

In this chapter, wheat flour obtained from 17 pure wheat cultivars was analyzed for a series of standard flour quality parameters, farinograph and alveograph rheological properties and by rotational rheometry.

An extensive correlation study revealed the complex interplay between flour composition, water absorption and dough rheological properties. Reducing these complex interactions to one or more parameter(s) which may predict breadmaking potential is the main reason why so many different methods for flour quality control have been developed.

For this data set, multiple linear regression showed that a model consisting of protein content, farinograph water absorption and alveograph P/L could best predict bread volume. This model could predict 80% of the variation in bread volume. So, it seems that the interaction between protein content, water absorption and dough extensibility largely determines the breadmaking

potential of the wheat flours. Through model validation with two wheat flours previously not included in the dataset, it was confirmed that this model was best suited for predicting the bread volume of these wheat flours. This model thus seems to offer the most potential for use in routine standard quality control of wheat flour. However, extending the dataset with more wheat flours would then be recommended.

The rheological parameter showing the highest linear correlation with bread volume was found to be phase angle delta obtained from the dynamic oscillation experiments. On its own, phase angle delta can predict almost 70% of the variation in bread volume in this set of wheat cultivars. It is thought that phase angle delta reflects the structural organization of the dough system as a result of the effective water distribution in the dough and the interactions existing between the different dough constituents.

With respect to rotational rheometry, a clear relationship was found between the small deformation dynamic oscillation measurements which are performed in the LVR and the creep-recovery measurements which fall outside the LVR. $J_{c,max}$ shows a more than proportional increase when the dynamic moduli decrease or phase angle delta increases. It has thus been shown that dough microstructure as measured by dynamic oscillation can be related to large deformation behaviour. This may be interesting for understanding dough behaviour during processing in which larger shear deformations occur.

$J_{c,max}$ may be used as a measure of dough strength or dough rigidity. Creep-recovery parameters were shown to relate to alveograph extensibility. However, dough preparation and formulation were different. Performing both rheological methods on dough samples of similar composition may elucidate if creep deformation can be related more closely to dough extensibility.

Applying the 6-parameter Burgers model was shown to be useful in grouping wheat cultivars with similar viscoelastic properties. Key parameters were the creep-recovery deformation behaviour, and the recovery retardation times, especially r_2. However, a sufficiently high stress is needed to group the wheat cultivars to similar rheological properties. It seems that the recovery retardation times better reflect differences in elasticity than the amount of recovery compliance.

4.7. Considerations for the following chapters

Some limitations were present in the setup of this chapter. First, dough preparation was fixed to one mixing time whereas every wheat flour has its optimal mixing requirements. Also rheological testing was performed on wheat flour-water doughs whereas in breadmaking other ingredients are incorporated into the dough which impact dough rheological properties and thus breadmaking potential. Therefore, it was decided to first investigate the effect of the mixing process and dough formulation on dough rheology and microstructure which will be discussed in more detail in Chapter 5.

In this chapter the main goal was to evaluate the use of rotational rheometry to assess the rheological properties of wheat flour dough and to relate the findings to breadmaking potential. However, as extensional deformations also play an important role in dough processing, from now on also uniaxial extension measurements will be included in the further rheological testing.

It was chosen to perform creep-recovery experiments at a shear stress of 250 Pa as this was more successful in grouping the wheat cultivars according to their rheological properties. As Burgers model parameters obtained from the creep phase highly correlated with $J_{c,max}$ it was decided to only model the recovery phase to obtain information about the recovery retardation times.

In this chapter, dynamic oscillation tests were in fact ‘single point’ measurements because they were obtained only at one frequency. From now on, frequency sweeps will be performed to investigate frequency dependence.

Chapter 5

Influence of the mixing process on dough rheology and microstructure

BROEID: in: -dat'n etj gien broeid-:
1. dat vergt geen onderhoud 2. er is geen haast bij

[Oilsjtersen Diksjoneir]

Relevant publication:

Van Bockstaele, F., De Leyn, I, Eeckhout, M., Meesen, G., Van Oostveldt, P. & Dewettinck, K. Influence of the mixing process on dough rheology and microstructure, Journal of Cereal Science, Submitted.

5.1. Introduction

The main goal of the mixing process is to obtain a homogeneous dough system which comprises of the appropriate rheological properties and a sufficient amount of aeration for subsequent processing. An optimally developed dough matrix should be able to retain the gas generated during fermentation in the form of numerous small gas cells. Furthermore, the dough matrix should have a proper balance between viscous flow and elastic strength so that the loaf can expand adequately during proofing and the early stages of baking, still retaining its rounded form (Stauffer, 2007). Dough rheological properties determine the behaviour of the dough pieces during mechanical handling such as dividing, rounding and molding. Further, rheological properties affect the quality of the finished loaf of bread (Bloksma and Bushuk, 1988). Thus, knowledge or characterization of the rheological properties of dough can be effective in predicting its behaviour during processing and controlling its quality (Ross et al., 2004).

Mixing significantly changes the rheological properties of wheat dough (Schluentz et al., 2000). Conflicting reports are available on the effect of mixing on the rheological properties of dough measured by small deformation dynamic oscillation. Some authors reported a decrease of the elastic modulus (G') and an increase in the loss tangent (tan δ), suggesting that the dough becomes less elastic with increasing mixing time (Dreese et al., 1988; Khatkar, 2004; Mani et al., 1992). Others only observed a decrease of the dynamic moduli after reaching a maximum at optimum dough development (Ross et al., 2004; Zheng et al., 2000) or noticed an increase of G' with increasing mixing time (Abdelrahman and Spies, 1986; Bohlin and Carlson, 1980). Also, a decrease (Kim et al., 2008) and an increase (Letang et al., 1999) in phase angle δ have been found with increasing mixing time. Finally, the effect of mixing on rheological properties strongly depends on the wheat type under study (Khatkar, 2004).

Also large deformation extensional measurements are frequently used to characterize dough rheology. From extension tests on wheat flour doughs developed in a mixograph it was found that after an initial plateau, both resistance to extension and extensibility decreased with increasing mixing time (Gras et al., 2000). Peighambardoust et al. (2006) observed that z-blade mixing initially led to a dough with higher fracture stress and stronger apparent strain hardening which decreased with further mixing. Flour type, mixing speed and work input

affected strain hardening index, failure stress and failure strain measured in biaxial extension (Chin and Campbell, 2005b).

5.2. Objective

Although the mixing process has been the subject of many previous research, the aim of this study was to investigate more in detail:

- the influence of mixing time or mixing energy input on dough microstructure and rheology
- the effect of mixer type (z-blade vs. pin mixer) on dough microstructure development
- the effect of dough formulation on dough microstructure and rheology (flour-water dough (FW) or baking formulation (BF) dough)

5.3. Research strategy

Doughs were prepared at different mixing times in two different dough mixers, more specifically a z-blade and a pin mixer, which are believed to exert a different type of deformation during dough development. Further, two types of dough formulation were investigated, more specifically a simple flour-water dough and the baking formulation dough which also contained salt and ascorbic acid at levels used in the breadmaking test.

Rheological properties of the mixed doughs were determined by means of rotational rheometry (dynamic oscillation and creep-recovery) and uniaxial extension.

To explain differences in dough rheology, dough microstructure was visualised by confocal scanning laser microscopy (CSLM).

Results will be first presented for the rheological methods, microstructure and breadmaking tests. This will be followed by a discussion which is divided into two parts (FW dough and BF dough).

5.4. Materials and Methods

5.4.1. Wheat flour

All experiments were performed with a standard bread flour (Epi B) obtained from Paniflower (Ghent, Belgium). Quality characteristics of Epi B flour are given in Table 5.1.

Table 5.1 Quality characteristics of Epi B wheat flour

Protein content (%dm)	11.9	*Farinograph*[*]	
Gluten index (%)	95	WA (%)	57.8
Wet gluten (%)	30.4	DDT (min)	3
Zeleny (mL)	40	Stability (min)	9.5
Ash content (%dm)	0.53	*Alveograph*[**]	
Damaged starch (%)	4.7	P (mmH_20)	96
Falling number (s)	315	L (mm)	89
		W (10^{-4} J)	287

[*]WA: water absorption; DDT: dough development time
[**] P: tenacity; L: extensibility; W: deformation energy

5.4.2. Mixing experiments

To investigate the effect of dough mixing on dough rheology and microstructure, two different mixing devices were used: a 10-g pin mixer and a 50-g farinograph mixer which will be referred to as the z-blade mixer. Both mixer types are shown in Figure 5.1. To be able to use the z-blade mixer bowl apart from the Farinograph apparatus, a special motor drive with speed control regulation was constructed. The mixer head was connected to a water bath to regulate the temperature and before experiments were started, the rotation speed of the motor was checked (63 rotations/min).

To be able to compare the two dough mixers, the energy input during mixing was recorded with a Fluke 43B Power Quality Analyzer. As the Fluke 43B measured the total amount of energy used by the mixer, the energy necessary for dough development was calculated by subtracting the energy needed when the mixer has no load from the energy needed when mixing the dough. The energy input is expressed as Joule per gram of dough in the mixer bowl (J/g dough).

Figure 5.1 10 g pin mixer (left) and farinograph 50 g mixer bowl (including adapted motor drive with speed regulation)

Experiments were performed on two dough formulations. The first mixture only consisted of flour and deionised water according to the farinograph water absorption and will be referred to as flour-water (FW) dough. The second mixture also contained 1.5% salt and 25 ppm ascorbic acid, which corresponds to the amounts used in the standard baking procedure and will be called the baking formulation (BF) dough. All mixing experiments were carried out at 25°C. Samples for rheological evaluation were taken at 1.5, 2, 3, 4, 6 and 8 min mixing for the pin mixer and at 2, 4, 6, 10 and 14 min for the z-blade mixer. At 8 min (pin) and 14 min (z-blade) mixing, the doughs became sticky which indicated overmixing.

5.4.3. Rheology

5.4.3.1. Rotational rheometry

Measurements were performed as described in Chapter 3 with following modifications.
Dough samples obtained from the pin or z-blade mixer rested for 10 min in a closed container at room temperature. A small piece was taken from the inner part of the dough and loaded between the parallel plates. During testing temperature was controlled at 25°C.

First, strain sweeps were performed to determine a single strain value which could be used for all the frequency sweeps. Strain sweeps were performed on dough developed in both mixer types at the shortest and longest mixing time and this at two frequencies (0.1 and 100 Hz). A strain value of 4.10^{-4} was selected which guaranteed that all measurements were made inside the linear viscoelastic region (LVR).

Further, frequency sweeps were performed on all dough samples. Frequency varied logarithmically from 0.1 to 100 Hz. Obtained parameters as function of frequency are the elastic modulus (G'), the viscous modulus (G") and the phase angle (δ). Reported values are the mean of four independent replicates (each representing a separately prepared dough).

After the frequency sweep, a creep-recovery measurement was performed. A shear stress of 250 Pa was put on the dough sample for 5 min, followed by a recovery phase of 10 min (shear stress = 0 Pa). The recovery phase was modeled using the Burgers model as described in Chapter 3.

5.4.3.2. Uniaxial extension

Measurements were performed as described in 2.2.4.3. Doughs rested during 10 min before transferring them to the Teflon mould for the formation of the dough strips.

5.4.4. Breadmaking tests and bread quality

Breadmaking tests were performed to evaluate the influence of mixing time on bread quality. The baking procedure was performed as described in 2.2.5. To obtain a sufficient amount of dough, the mixers were scaled up to a 300 g farinograph (Brabender, Germany) and a Majorpin mixer (Henry Simon Ltd, UK). As the rotation speed of the majorpin mixer (66 rpm) was lower than the 10 g pin mixer (92.5 rpm) and the speed could not be changed, mixing times were adapted to obtain the same level of dough development since it has been shown that mixing in a pin mixer is greatly rate-independent (Anderssen et al., 1998).

5.5. Results

5.5.1. Dough development

To illustrate the dough development of the Epi-B FW dough in the pin mixer, the mixogram of the flour-water mixture is shown in Figure 5.2A. A peak in dough resistance can be seen between 2 and 3 min of mixing and the dough stays relatively stable under prolonged mixing. The farinogram shown in Figure 5.2B demonstrates the dough development of the FW mixture in the z-blade mixer. The curve was recorded at 25°C as this temperature was used

for the dough preparation for the rheological testing. It can be seen that the FW mixture shows a high stability during mixing.

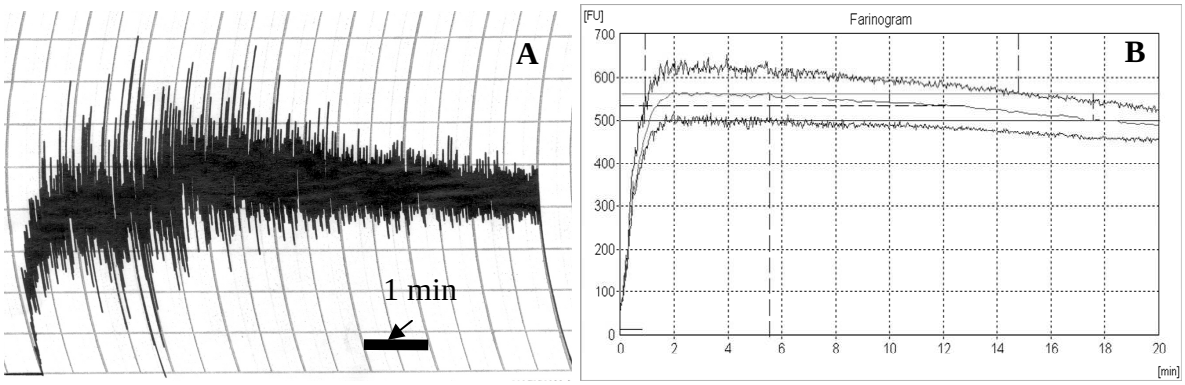

Figure 5.2 Mixogram (A) and farinogram (B) of Epi B flour-water dough

To be able to compare the two mixer types, the energy input during mixing was recorded for the FW and BF doughs. Figure 5.3 shows the cumulative energy input as function of the mixing time. For the pin mixer, a similar energy input was recorded for both formulations. For the z-blade mixer, mixing of the BF dough surprisingly required a lower energy input in comparison with the FW dough. The farinogram of the BF dough is shown in Figure 5.4. It can be seen that the consistency of the BF dough is slightly lower compared to the FW dough, so this may explain the lower energy required for mixing the BF dough. On the other hand, the BF dough shows a considerably longer stability during mixing indicating increased dough strength.

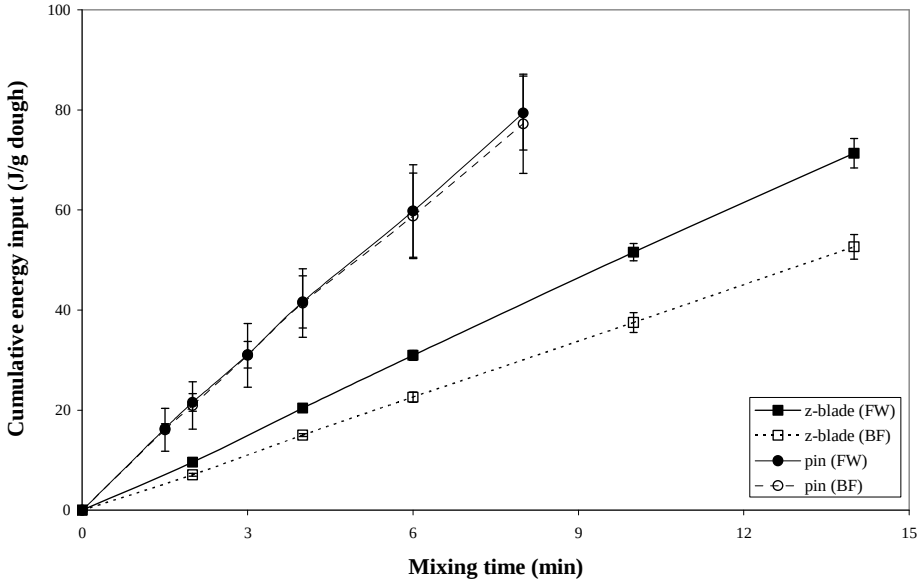

Figure 5.3 Cumulative energy input during dough mixing of the flour-water (FW) and baking formulation (BF) dough in a pin and z-blade mixer

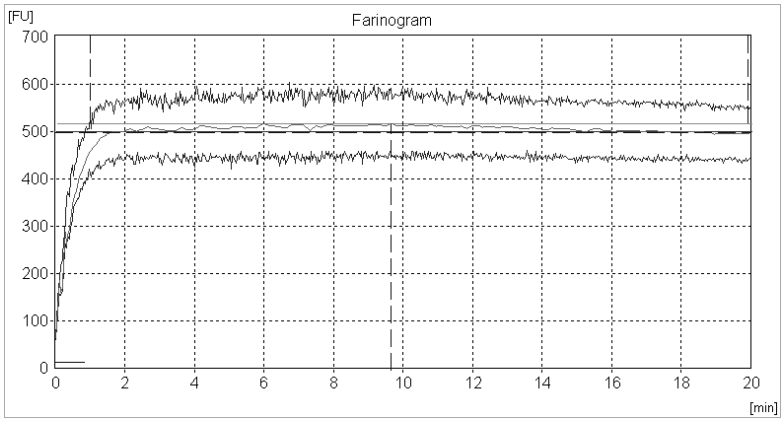

Figure 5.4 Farinogram of the baking formulation dough recorded at 25°C

5.5.2. Dough rheology

5.5.2.1. Dynamic oscillation

Frequency sweeps were performed to investigate the effect of the mixing process on the small deformation dynamic oscillation properties of the dough. From the frequency sweeps, frequencies of 1 and 10 Hz were selected to compare the values for G' and G" for different mixing times. The results are summarized in Table 5.2 according to mixer type and dough formulation.

It can be observed that mixing time has a limited influence on G' and G". Only for the FW dough developed in the pin mixer, a maximum can be observed for G' and G" after 3 min mixing. It can also be noted that mixer type has little effect on the elastic and viscous moduli. A significant difference was observed for G' of the FW dough between pin mixer and z-blade mixer at the maximum mixing times. G' and G" only show a slight increase when salt and AA are added to the dough formulation.

Figure 5.5 shows the evolution of phase angle delta as function of frequency for the two mixer types and the two dough formulations. In general, δ increases with increasing frequency which means that the doughs behave less elastic when the strain is applied faster. However, the doughs retain their predominant elastic character as δ does not exceed 30°. For the FW dough, δ decreases significantly with increasing mixing time for both mixer types. However, this is only true at low frequencies. Adding salt and ascorbic acid to the dough formulation, causes a decrease in δ which then stays relatively stable during the mixing process. Only

overmixing in the pin mixer (8 min), causes an increase in phase angle δ which indicates loss of elastic bonds.

In conclusion, it can be stated that in contrast with G' and G", which are only slightly affected by the mixing process, differences are more pronounced when looking at the phase angle. A clear influence of mixing time and dough formulation can be observed whereas mixer type has little impact.

Table 5.2 Elastic (G') and viscous (G") moduli of FW and BF doughs developed in a pin mixer or a z-blade mixer for different mixing times (MT)[*]

	FW dough				*BF dough*			
MT	*1 Hz*		*10 Hz*		*1 Hz*		*10 Hz*	
(min)	*G'*	*G"*	*G'*	*G"*	*G'*	*G"*	*G'*	*G"*
Pin mixer								
1.5	8475 ^{a}	3193^{a,b}	14243 ^{a}	6192 ^{a}	10105 ^{a}	3418 ^{a}	16098 ^{a}	6536 ^{a}
2	9321 ^{a,b}	3471^{a,b}	15583 ^{a,b}	6699 ^{a}	10455 ^{a}	3551 ^{a}	16720 ^{a}	6790 ^{a}
3	10001 ^{b}	3623 ^{a}	16555 ^{b}	6699 ^{a}	10394 ^{a}	3541 ^{a}	16740 ^{a}	6790 ^{a}
4	9666 ^{a,b}	3439 ^{a,b}	15865 ^{a,b}	6665 ^{a}	10130 ^{a}	3444 ^{a}	16323 ^{a}	6684 ^{a}
6	9041 ^{a,b}	3105 ^{b}	14598 ^{a,b}	6021 ^{a}	10058 ^{a}	3423 ^{a}	16223 ^{a}	6563 ^{a}
8	8823 ^{a,b}	3078 ^{b}	14410 ^{a,b}	5971 ^{a}	8989 ^{a}	3178 ^{a}	14683 ^{a}	6038 ^{a}
Z-blade mixer								
2	9455 ^{a}	3528 ^{a}	15768 ^{a}	6742 ^{a}	10402 ^{a}	3459 ^{a}	16418 ^{a}	6556 ^{a}
4	9939 ^{a}	3694 ^{a}	16573 ^{a}	7181 ^{a}	9519 ^{a}	3229 ^{a}	15325 ^{a}	6244 ^{a}
6	9620 ^{a}	3557 ^{a}	15908 ^{a}	7181 ^{a}	10813 ^{a}	3662 ^{a}	17385 ^{a}	6244 ^{a}
10	10031 ^{a}	3559 ^{a}	16460 ^{a}	6990 ^{a}	10047 ^{a}	3443 ^{a}	16220 ^{a}	6625 ^{a}
14	10033 ^{a}	3473 ^{a}	16368 ^{a}	6831 ^{a}	10242 ^{a}	3548 ^{a}	16678 ^{a}	6822 ^{a}

[*] Values indicated with different letters in the same column are significantly different (α=0.05)

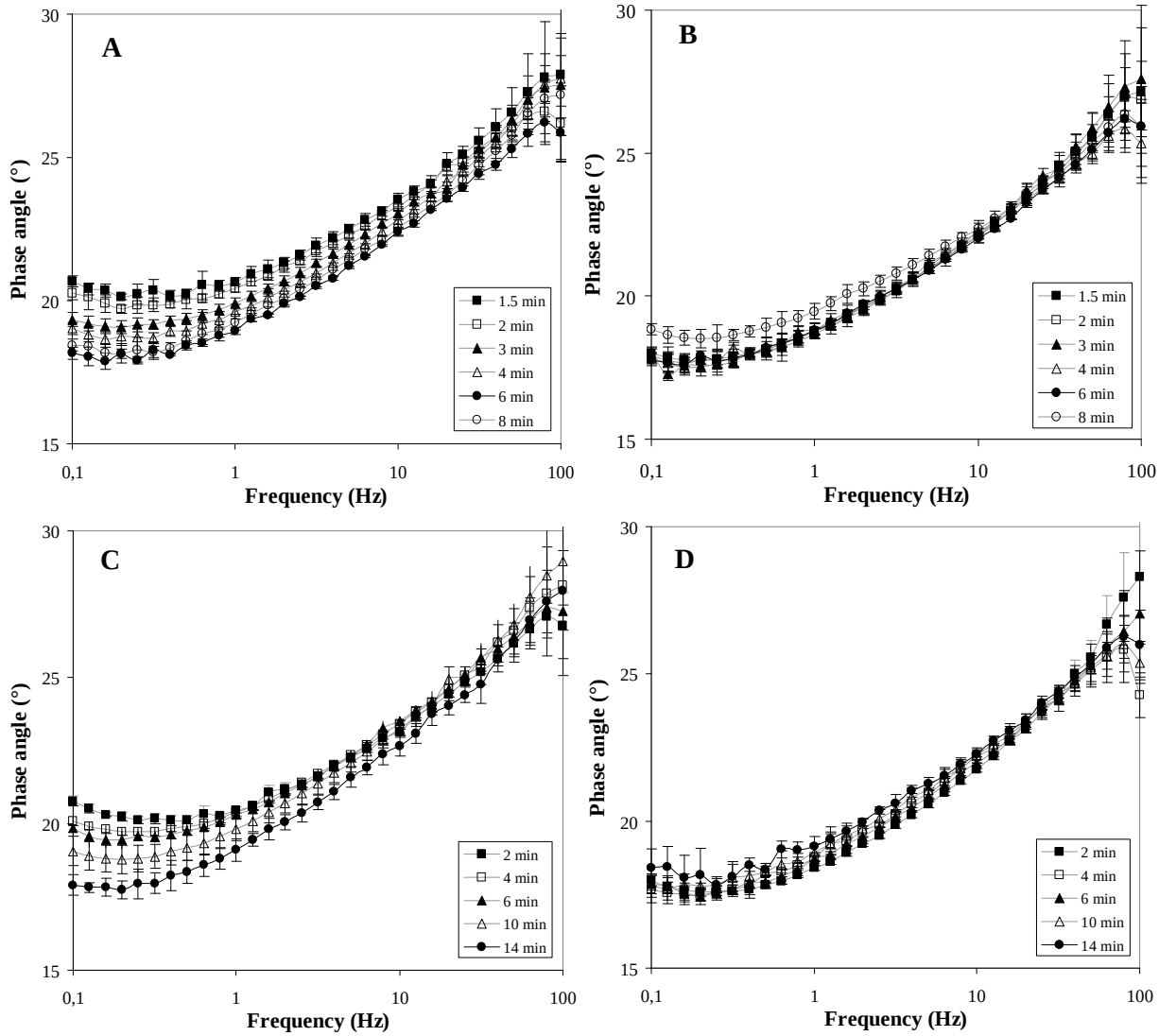

Figure 5.5 Phase angle δ as function of frequency for doughs developed in a pin mixer (A: FW dough; B: BF dough) and a z-blade mixer (C: FW dough; D: BF dough). Dough samples were analyzed at different mixing times.

5.5.2.2. Creep-recovery

In the creep-recovery measurements, a shear stress is put on the dough samples which evokes a deformation beyond the LVR. After the deformation, the elastic recovery of the dough is recorded. In Figure 5.6 the maximum creep compliance, $J_{c,max}$, is plotted as function of the energy input for both dough formulations during mixing in the pin mixer (A) and the z-blade mixer (B).

When the FW dough is mixed in the pin mixer, a clear decrease in $J_{c,max}$ of the mixed doughs is observed. Increased mixing makes the FW dough less deformable under a constant shear stress, indicating increased dough strength.

At the start of mixing, the BF dough already shows a low $J_{c,max}$. The addition of salt and ascorbic acid has a pronounced effect on dough strength already at short mixing times. No significant effects are seen during further mixing, indicating dough stability.

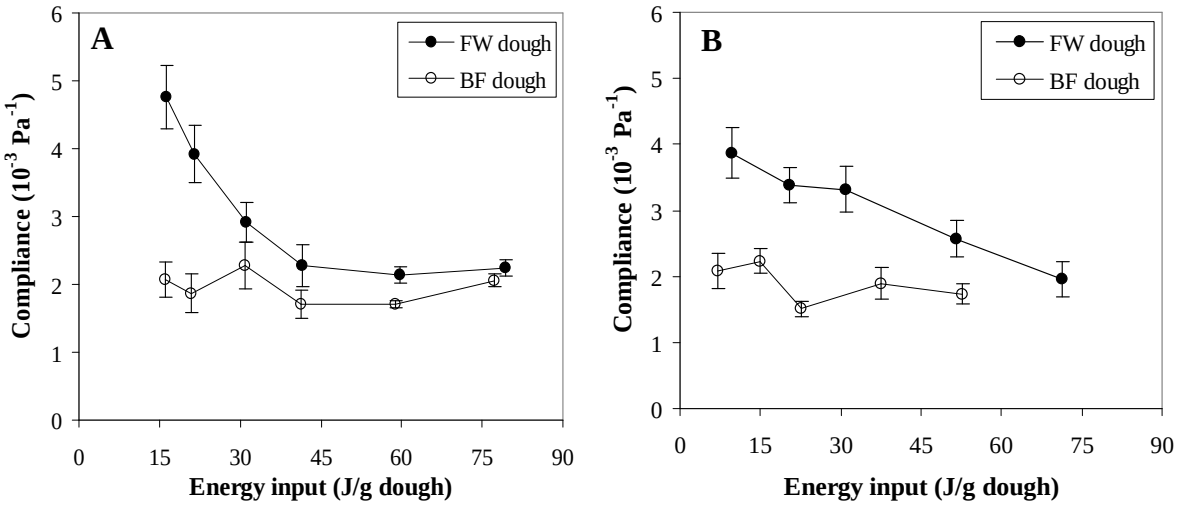

Figure 5.6 Maximum creep compliance ($J_{c.max}$) as function of increasing energy input during dough development in a pin mixer (A) and a z-blade mixer (B) for two dough formulations (FW and BF dough)

For the FW dough in the z-blade mixer, a different pattern can be observed. $J_{c,max}$ stays relatively stable up to an energy input of 30 J/g dough to decrease at higher energy inputs. For the BF dough, also a much lower $J_{c,max}$ is found compared to the FW dough. A minimum seems to be reached after a mixing time of 6 minutes (energy input of 22.6 J/g dough) which may be an indication of maximum dough strength.

It was found that the parameters obtained from the Burgers model which was applied to the creep data, were highly positively correlated with $J_{c,max}$ and thus showed the same trends due to mixing as seen for $J_{c,max}$. Steady state viscosity μ_0, on the other hand, showed to be inversely related with $J_{c,max}$. Therefore, results are shown in detail in Figure 5.7. Steady state viscosity of the doughs is presented as function of the energy input for both dough formulations during mixing in the pin mixer (A) and the z-blade mixer (B). For the FW doughs, the increase of μ_0 with increasing energy input is dependent on mixer type. This mixer dependency was also seen in the results of $J_{c,max}$. The BF dough shows a higher steady state viscosity compared to the FW dough. For the z-blade mixer a significant increase is

observed in steady state viscosity between 4 and 6 min mixing. This may indicate that the dough has reached maximum dough strength.

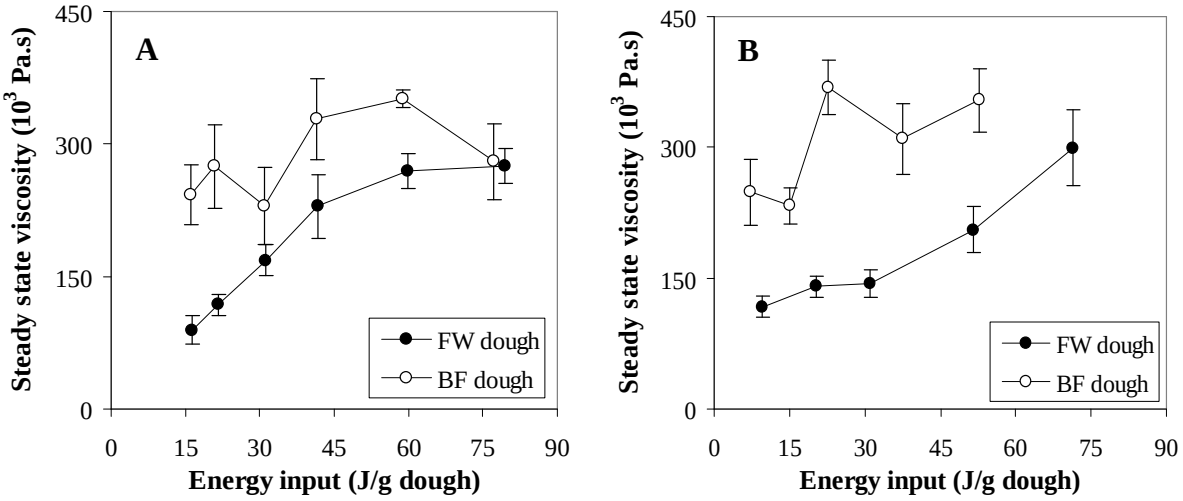

Figure 5.7 Steady state viscosity μ_0 as function of increasing energy input during dough development in a a pin mixer (A) and a z-blade mixer (B) for two dough formulations (FW and BF dough)

The recovery characteristics are summarized in Table 5.3. Relevant parameters are the maximum recovery compliance ($J_{r,max}$), the percentage recovery which expresses $J_{r,max}$ as a percentage of the maximum creep deformation ($J_{c,max}$) and the retardation times (r_1, r_2) of which higher values indicate a slower recovery.

Remarkably, $J_{r,max}$ remains constant during the mixing process and is not influenced by mixer type. Only a higher $J_{r,max}$ is observed for FW dough at 1.5 min mixing in the pin mixer. This is probably related to the higher creep compliance also found at this point. The constant $J_{r,max}$ is seen for both dough formulations, however lower recoveries are found for the BF dough.

For the FW doughs, a gradual increase in percentage recovery is observed, mainly due to the decrease of $J_{c,max}$ with increasing mixing time. Parallel, a significant decrease of the retardation times is observed, which indicates a faster recovery and thus a more elastic gluten network. In the pin mixer, FW doughs reach maximum dough strength after a mixing time of 6 minutes (r_2=42.3s). In the z-blade mixer maximum dough strength is observed at a mixing time of 14 min (r_2=42.9s). At this point the doughs from both mixers show similar rheological properties.

Table 5.3 Recovery properties of flour-water (FW) and baking formulation (BF) developed in a pin mixer and a z-blade mixer at different mixing times: total recovery compliance $J_{r,max}$, percentage recovery ($J_{r,max}/J_{c,max}$*100) and retardation times r_1 and r_2[*]

	FW dough				*BF dough*			
MT (min)	$J_{r,max}$ (10^{-3} Pa^{-1})	%recovery	r_1 (s)	r_2 (s)	$J_{r,max}$ (10^{-3} Pa^{-1})	%recovery	r_1 (s)	r_2 (s)
Pin mixer								
1.5	1.65 ± 0.13^{a}	33.6 ± 1.8^{a}	1.7 ± 0.1^{a}	51.8 ± 2.0^{a,b}	1.18 ± 0.11^{a}	57.2 ± 2.0^{a}	1.2 ± 0.1^{a,b}	42.3 ± 0.8^{a}
2	1.45 ± 0.09^{a,b}	37.2 ± 1.7^{a}	1.7 ± 0.1^{a}	53.6 ± 1.2^{a}	1.12 ± 0.15^{a}	60.1 ± 1.3$^{a.b}$	1.1 ± 0.1$^{a.b}$	41.5 ± 1.0^{a}
3	1.34 ± 0.07^{b}	46.2 ± 2.3^{b}	1.6 ± 0.1^{b}	50.9 ± 0.8^{b}	1.41 ± 0.17^{a}	62.2 ± 2.3$^{b.d}$	1.2 ± 0.1^{a}	41.6 ± 0.7^{a}
4	1.33 ± 0.22^{b}	54.1 ± 3.6^{c}	1.3 ± 0.1^{c}	45.8 ± 1.1^{c}	1.13 ± 0.13^{a}	66.6 ± 0.8^{c}	1.1 ± 0.1^{b}	39.2 ± 0.6^{b}
6	1.3 ± 0.05^{b}	60.7 ± 1.1^{d}	1.2 ± 0.1^{d}	42.3 ± 0.3^{d}	1.15 ± 0.04^{a}	67.5 ± 0.6^{c}	1.1 ± 0.1^{b}	39.1 ± 0.4^{b}
8	1.36 ± 0.12^{b}	63.4 ± 1.3^{d}	1.3 ± 0.1$^{c.d}$	43.8 ± 1.4$^{c.d}$	1.35 ± 0.04^{a}	65.5 ± 1.0$^{c.d}$	1.3 ± 0.1^{a}	42.5 ± 0.9^{a}
Z-blade mixer								
2	1.42 ± 0.11^{a}	36.8 ± 1.6^{a}	1.8 ± 0.1^{a}	55.9 ± 1.4^{a}	1.13 ± 0.09^{a}	54.7 ± 2.8^{a}	1.3 ± 0.1^{a}	42.6 ± 0.4^{a}
4	1.38 ± 0.05^{a}	41.0 ± 2.0$^{a.b}$	1.7 ± 0.1^{a}	53.6 ± 0.5^{b}	1.28 ± 0.06^{a}	57.6 ± 2.8^{a}	1.4 ± 0.1^{a}	42.8 ± 0.4^{a}
6	1.39 ± 0.08^{a}	42.1 ± 2.3^{b}	1.6 ± 0.1^{b}	50.2 ± 0.6^{c}	0.98 ± 0.07^{a}	64.1 ± 0.9^{b}	1.2 ± 0.1^{a}	42.4 ± 0.4^{a}
10	1.31 ± 0.07^{a}	51.4 ± 3.3^{c}	1.4 ± 0.1^{c}	47.0 ± 1.3^{d}	1.21 ± 0.13^{a}	63.8 ± 1.2^{a,b}	1.3 ± 0.1^{a}	42.9 ± 0.9^{a}
14	1.20 ± 0.14^{a}	61.4 ± 1.5^{d}	1.2 ± 0.1^{d}	42.9 ± 0.8^{e}	1.25 ± 0.23^{a}	66.0 ± 0.5^{b}	1.2 ± 0.1^{a}	41.5 ± 1.4^{a}

[*] Values indicated with different letters in the same column of one mixer type are significantly different (α=0.05)

In case of the BF dough, the percentage recovery shows a significant increase between 3 and 4 min mixing for the pin mixer and between 4 and 6 min mixing for the z-blade mixer. This may indicate the point of optimal development of the dough matrix which is maintained upon further mixing. At this point, dough developed in the pin mixer, has a significantly higher percentage recovery and lower retardation times compared to the z-blade mixer. In general, retardation times are lower for the BF dough compared to the FW dough and remain relatively stable during the mixing process. This may indicate that elastic properties are already present after relatively short mixing times.

5.5.2.3. Uniaxial extension

The influence of the mixing process on uniaxial extension properties of the dough was investigated. In an extension measurement, the dough is subjected to a large deformation, namely an uniaxial extension until dough rupture. Parameters obtained are resistance to extension (R_{max}), extensibility (Ext) and area under the curve (Area). These measurements give information about the ability of the dough to withstand large extensional deformations occurring during breadmaking.

Figure 5.8 shows R_{max}, Ext and Area as function of increasing energy input during dough development of FW and BF dough in the pin mixer and the z-blade mixer.

Concerning the FW dough, similar results were obtained for both mixer types. R_{max} and Ext remain constant in the beginning of dough development. But when the work input exceeds 40 J/g dough, an increase of R_{max} and a decrease of Ext can be seen. This causes Area to decrease during mixing.

BF dough shows a much higher strength when extended compared to the FW dough. In Figure 5.8 it can be seen that dough reaches a maximum in dough strength during the mixing process. The maximum dough strength is observed as a maximum in R_{max} and Area. At the same moment a decrease in Ext is found.

Similar trends were found for BF dough mixed in a pin mixer and a z-blade mixer. However, the maximum dough strength in the pin mixer was reached at higher energy input and at that point a higher dough strength (Area) was observed compared to the z-blade mixer.

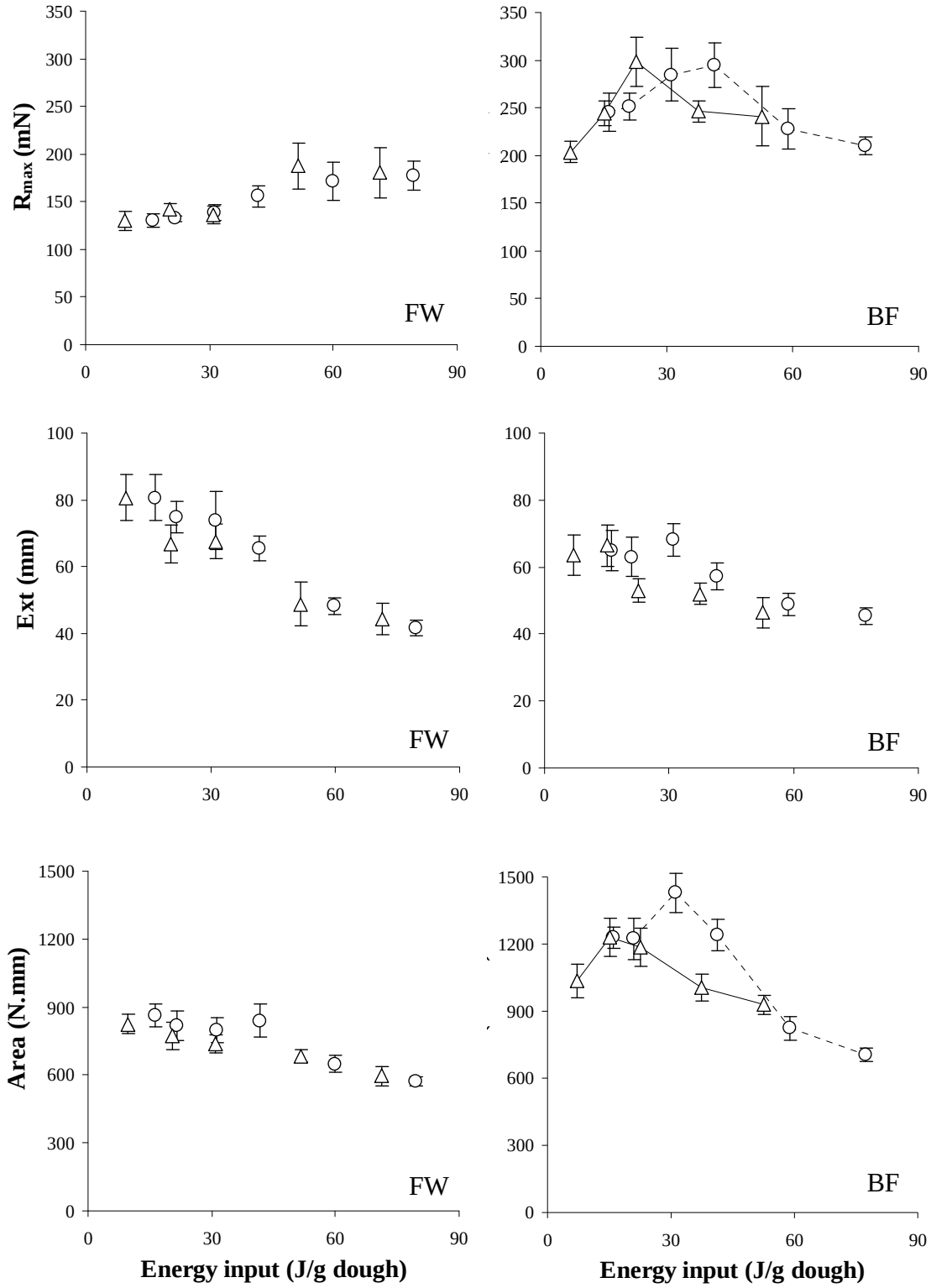

Figure 5.8 Resistance to extension (R_{max}), extensibility (Ext) and area under the curve (Area) of flour-water (FW) and baking formulation (BF) dough as function of the energy input during dough development in a pin mixer (○) and a z-blade mixer (Δ).

From the force-displacement curves, uniaxial extension fracture properties were calculated (Table 5.4). In case of FW dough, fracture stress and strain show a decrease only after longer mixing times whereas strain hardening index increases.

For the BF dough, a maximum in fracture stress can be observed for both mixers. However, maximum fracture stress in the z-blade mixer is significantly lower compared to the pin mixer. Also, the maximum fracture stress does not coincide with the observed maximum in R_{max} as was also previously observed for Area. Fracture strain of the baking formulation is similar to the FW dough. On the other hand, strain hardening index is significantly higher in comparison with the FW dough and a minimum energy input seems to be required to obtain the maximum value in both mixer types.

Table 5.4 Dough fracture properties calculated from uniaxial extension of flour-water (FW) dough and baking formulation (BF) dough developed in a pin mixer and a z-blade mixer at different mixing times (MT) [*]

	FW dough			*BF dough*		
MT (min)	σ_{max} (kPa)	ε_H (-)	SHI (-)	σ_{max} (kPa)	ε_H (-)	SHI (-)
Pin mixer						
1.5	34.6 ± 1.9^{a}	2.3 ± 0.1^{a,b}	1.28 ± 0.04^{a,b}	52.0 ± 1.8^{a}	2.1 ± 0.1^{a,b}	1.39 ± 0.03^{a}
2	33.5 ± 2.2^{a}	2.3 ± 0.1^{b}	1.27 ± 0.03^{a}	53.6 ± 4.4^{a}	2.1 ± 0.1^{a,b}	1.44 ± 0.03^{b}
3	34.3 ± 2.3^{a}	2.2 ± 0.1^{b}	1.35 ± 0.03^{c}	64.3 ± 3.2^{b}	2.2 ± 0.1^{a}	1.53 ± 0.04^{c}
4	34.4 ± 2.8^{a}	2.1 ± 0.1^{a}	1.32 ± 0.03^{a,b,c}	56.8 ± 3.3^{a}	2.0 ± 0.1^{b}	1.52 ± 0.03^{c}
6	26.9 ± 1.7^{b}	1.8 ± 0.1^{c}	1.35 ± 0.04^{b,c}	36.5 ± 2.0^{c}	1.8 ± 0.1^{c}	1.52 ± 0.02^{c}
8	23.7 ± 0.8^{c}	1.7 ± 0.1^{d}	1.44 ± 0.03^{d}	30.1 ± 1.0^{d}	1.7 ± 0.1^{c}	1.53 ± 0.03^{c}
Z-blade mixer						
2	33.6 ± 1.9^{a}	2.3 ± 0.1^{a}	1.28 ± 0.02^{a}	42.0 ± 3.5^{a}	2.1 ± 0.1^{a,b}	1.37 ± 0.05^{a}
4	31.4 ± 2.3^{a}	2.1 ± 0.1^{b}	1.32 ± 0.02^{b}	52.6 ± 4.2^{b}	2.1 ± 0.1^{b}	1.45 ± 0.03^{b}
6	30.5 ± 1.7^{a,b}	2.2 ± 0.1^{b}	1.34 ± 0.03^{b,c}	50.0 ± 4.5^{b,c}	1.8 ± 0.1^{c}	1.51 ± 0.06^{b,c}
10	29.3 ± 1.0^{b}	1.8 ± 0.1^{c}	1.44 ± 0.07^{c,d}	43.6 ± 2.2^{a,c}	1.9 ± 0.1^{a,c}	1.51 ± 0.04^{b,c}
14	25.1 ± 1.5^{c}	1.7 ± 0.1^{c}	1.45 ± 0.05^{d}	41.4 ± 1.9^{a}	1.7 ± 0.1^{c}	1.61 ± 0.07^{c}

[*] Values indicated with different letters in the same column of one mixer type are significantly different (α=0.05)

5.5.3. Dough microstructure

To be able to understand changes in rheology due to the mixing process, confocal scanning laser microscopy (CSLM) was used to visualize the dough microstructure. CSLM images of

doughs developed in the pin mixer and the z-blade mixer are shown in Figure 5.9 and Figure 5.10 respectively. Images of FW and BF dough are presented at two different mixing times: 3 and 6 min for pin mixer and 6 and 14 min for the z-blade mixer.

FW dough developed in the pin mixer initially shows a network of fine gluten threads that surround the starch granules and which evolves into a more homogenous gluten phase upon prolonged mixing. For the BF dough, the gluten phase seems to be highly structured as longer and broader gluten strands are visible compared to the FW dough. This structure remains stable under prolonged mixing.

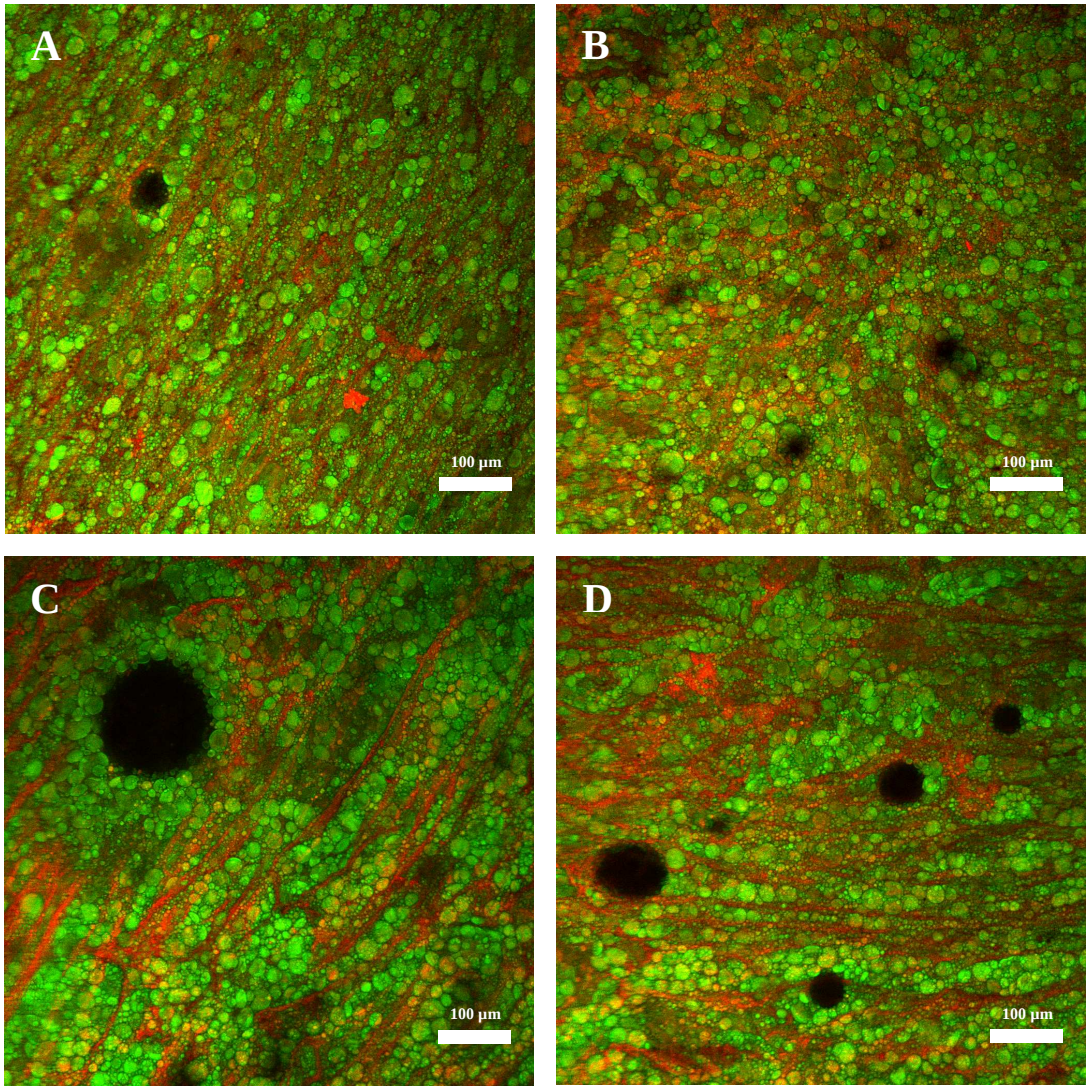

Figure 5.9 CSLM images of doughs developed in a pin mixer. A: FW dough - 3 min; B: FW dough - 6 min; C: BF dough - 3 min; D: BF dough - 6 min (green=starch granules, red=protein network, bar=100 µm)

FW dough developed in the z-blade mixer (Figure 5.10), initially shows a finely dispersed protein network. Upon further mixing, the proteins seem to form more aggregated structures. This is in contrast with Peighambardoust et al. (2006) who observed that a more homogeneous gluten phase was formed with increased mixing. However, their dough did contain 2% of salt and much longer mixing times were used. In the BF dough, broader gluten strands can be observed which appear somewhat more dispersed upon further mixing. However, the very long gluten strands as observed in the pin mixer are less present in the BF dough developed in the z-blade mixer.

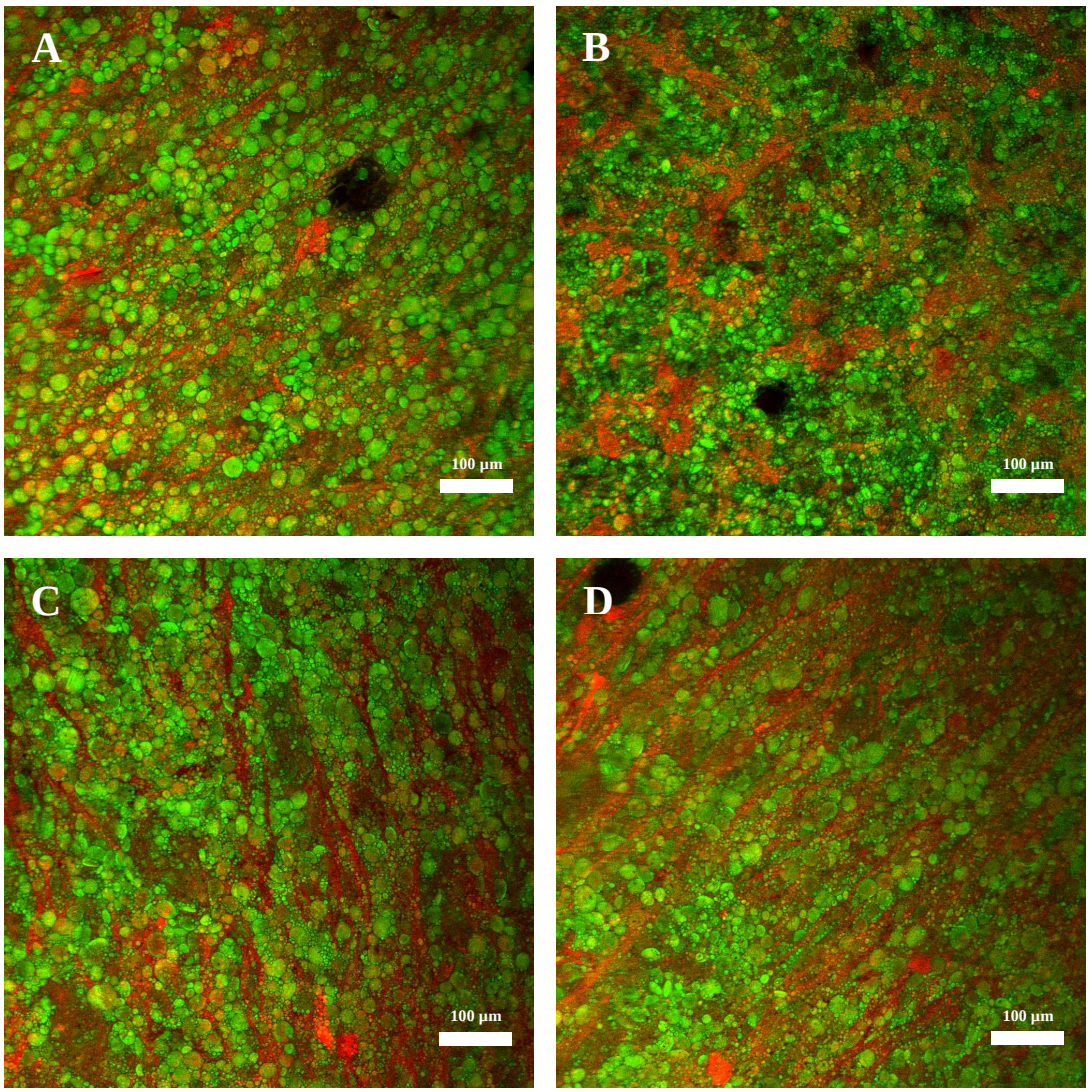

Figure 5.10 CSLM images of doughs developed in a z-blade mixer. A: FW dough - 6 min; B: FW dough - 14 min; C: BF dough - 6 min; D: BF dough - 14 min (green=starch granules, red=protein network, bar=100 µm)

5.5.4. Breadmaking tests

Breadmaking tests were performed with BF doughs including yeast and which were mixed for different mixing times in the pin mixer and the z-blade mixer. This allowed to evaluate the effect of mixing time and mixer type on bread quality. Both pan breads and plate breads were evaluated. The results are summarized in Annex 6.

For the pin mixer, bread volumes increased up to a mixing time of 4 min for pan bread and 6 minutes for plate bread. Higher mixing times did not negatively affect baking quality. This may be explained by the results of Don et al. (2005) in which it was shown that even after overmixing, the gluten structure can be restored during dough rest. Oven rise and the form ratio (H/W) were not significantly affected by mixing time. Bread weight slightly decreased with longer mixing times, probably due to the higher volumes obtained which enhances water evaporation during baking.

For the z-blade mixer, bread volumes increased up to a mixing time of 4 min for pan bread and 6 minutes for plate bread. A mixing time of 4 minutes was observed to obtain maximum oven rise and form ratio (H/W). Bread weight decreased significantly with increasing mixing time for pan bread. This was not observed for plate bread.

In general, no major differences in baking quality were found between the pin mixer and the z-blade mixer.

5.6. Discussion

5.6.1. FW dough

The rheological measurements performed on FW dough sampled during dough development, showed differences in dough development between the two mixer types. It is believed that different deformation occurs in pin and z-blade mixing. Pin mixers prepare dough with a pull-tear type, extensional mixing action (Gras et al., 2000). Z-blade mixers appear to form the dough with a gentle kneading or shearing action in which the dough is squeezed between the mixer blade and the mixer body (Haraszi et al., 2008). It has been shown that a z-blade mixer

provides a combination of rotational, shear and extensional deformations whereas dough in a pin mixer is mainly subjected to shearing and elongation (Jongen et al., 2003).
Dynamic oscillation showed a difference between doughs developed in the two mixer types. For doughs developed in the pin mixer, an optimum for G' and G" was found between 3 and 4 min of mixing (Table 5.2). This corresponds with the maximum in consistency as seen in the mixogram (Figure 5.2). Mixing beyond this optimum caused a drop in G' and G". This was not observed in the z-blade mixer where G' and G" remained constant even at a mixing time of 14 min (Table 5.2). However, the longest mixing times provided a comparable energy input to the dough in both mixers (Figure 5.3). So, the differences in dynamic moduli can probably be attributed to the deformation conditions in the mixers and this may be related to a different microstructure as visualised by CSLM. Haraszi et al. (2008) reported that the mixing action in a pin mixer breaks down the glutenin polymers and aggregates to a larger extent than the z-blade mixer.

On the other hand, a significant decrease of phase angle delta during dough development occurred for both mixers. It thus seems that FW dough becomes more elastic during mixing. Similar results were reported by (Kim et al., 2008) for hard wheat flour dough. The decreasing δ may be an indication of the formation of bonds which are able to store the energy during deformation. Both covalent and non-covalent bonds play a role in dough formation and development of which disulfide bonds play a key role in the interactions in doughs (Bushuk, 1998). On the molecular level, glutenin molecules are crucial in dough development. During dough development, glutenin disaggregates from its highly compact structure in the protein bodies to a more open structure in dough (Bushuk, 1998). During mixing, high molecular weight glutenins break into smaller units. The broken disulfide bonds reform between adjacent molecules that have been aligned along the lines of stress in the dough (Stauffer, 2007). Don et al. (2003b) showed that mixing decreased the amount of glutenin macro polymer (GMP) and no GMP fraction could be recovered at peak dough development indicating gluten network formation. Optimally mixed doughs also showed the highest relaxation half times which are a measure of dough elasticity and overmixing did not cause large changes (Don et al., 2005).

Together with the small deformation measurements, which are performed in the linear viscoelastic region, also creep-recovery measurements were carried out which resulted in deformations outside the LVR. It was found that $J_{c,max}$ decreased during dough development

but this was related to the mixer type (Figure 5.6) and lower retardation times were observed with increased mixing time. This also indicates the presence of more elastic bonds between the structural elements in the dough matrix which results in increased dough strength and the ability to recover faster when the stress is removed.

Figure 5.11 shows the relation between δ and $J_{c,max}$ for the data obtained from the mixing experiments of FW dough in both mixers. It appears that viscoelastic properties measured at small deformation can be related to the deformation occurring when a larger stress is applied.

A similar relationship has been found previously for FW doughs obtained from different wheat cultivars (4.5.3.4). Doughs which behave more elastic under small deformation, deform less when larger shear stress is applied. This means that rheological properties measured at small deformation may be useful in predicting dough performance in processing where larger stresses or strains are involved.

In contrast with the results obtained from rheometry, uniaxial extension properties of the FW dough were less clearly influenced by mixing. An increase in R_{max} and a decrease in Ext were seen during prolonged mixing. This may thus reflect the increased development of the gluten structure, lowering extensibility and increasing dough strength. Dough fracture properties show that the strain hardening index increases with mixing, suggesting a more interconnected gluten network which, at the longest mixing times, results in lower stress values at dough fracture due to the reduced extensibility of the dough.

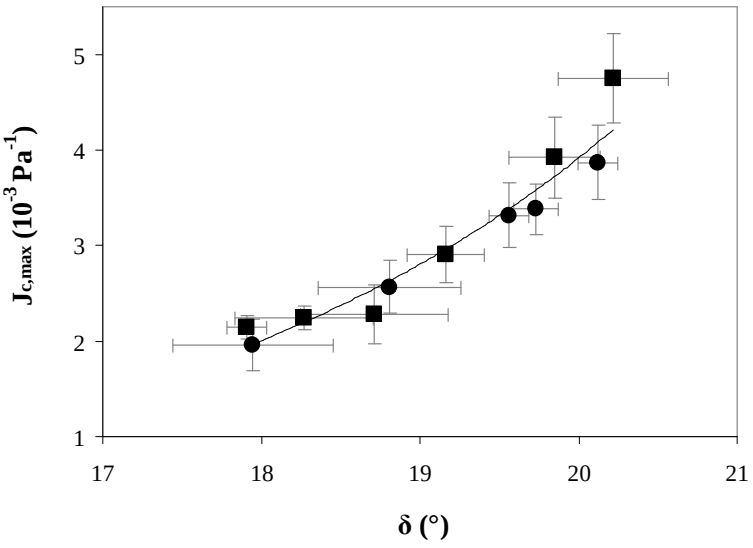

Figure 5.11 Relation between phase angle δ and the maximum creep compliance $J_{c,max}$. Data obtained from FW doughs at different mixing times in a pin mixer (squares) and a z-blade mixer (rounds). Error bars indicate standard deviation on mean value.

5.6.2. BF dough

In wheat flour quality control, rheological properties are usually determined of a flour-water mixture. However, in breadmaking other ingredients are added which may have a large impact on dough microstructure and rheology. In this study, 1.5% salt and 25 ppm ascorbic acid (AA) were added to the FW dough to simulate the formulation used in breadmaking.
Salt is one of the essential ingredients in bread which stabilizes yeast fermentation rate, enhances flavour and strengthens the dough (Miller and Hoseney, 2008). Ascorbic acid acts as an oxidizing agent by the formation of disulfide bonds between gluten proteins resulting in increased dough strength (De Leyn, 2006). Several mechanisms for the improving effect of AA have been proposed (Joye et al., 2009).

By adding salt and AA, the complexity of the FW dough increased as both chemical and physical interactions were added to the dough system. Incorporation of salt and AA to the dough formulation, had a large impact on dough rheology and microstructure. BF dough shows a more structured gluten network with thicker and longer gluten strands which remain stable during mixing. Differences in microstructure were observed between mixer types. The gluten strands of dough developed in the pin mixer appeared to be thinner and longer compared to the z-blade mixer indicating that mixing action influences microstructure formation.

BF dough showed a high stability during mixing which was reflected in stable values found for G', G", δ, $J_{c,max}$, $J_{r,max}$, r_1 and r_2. The dynamic moduli, G' and G", barely differed from the FW dough. Probably this is the result of the combined effect of salt and AA. An increasing salt concentration has been shown to cause a decrease in G' and G" (Lynch et al., 2009; Salvador et al., 2006) whereas AA causes an increase of the dynamic moduli (Berland and Launay, 1995; Miller and Hoseney, 1999). Van der Zalm et al. (2010) didn't observe differences in moduli in the range of 0-4 w% salt, most probably caused by the changes in water absorption to obtain constant dough consistency.
Values for δ were low from the start of the mixing process (±17.5° at 0.25Hz) in both mixers and were significantly different from the FW dough. The low δ indicates an increased cross-linking which is supported by the CSLM images. A decrease of phase angle delta has been reported upon addition of AA (Kenny et al., 1999), however this was not observed by others

(Berland and Launay, 1995; Miller and Hoseney, 1999). Salt has little effect on δ (Lynch et al., 2009; van der Zalm et al., 2010) or causes a decrease (Wehrle et al., 1997).

During dough development of BF dough, a maximum in dough strength was observed. Maximum dough strength corresponds to a maximum in R_{max} after 4 min (pin) and 6 min (z-blade) mixing. At this point also a decrease in Ext was observed. From the fracture properties, it can be seen that maximum σ_{max} occurs before the maximum in R_{max} is observed (3 min for pin and 4 min for z-blade). At this point also the maximum SHI is reached which remains stable upon further mixing. Maximum dough strength was also observed in the creep-recovery measurements by %recovery indicating that the elastic properties are fully developed.

Dough development of the BF dough was affected by mixer type. First, a lower energy input was required for dough development in the z-blade mixer. This means that the addition of salt and AA has a different effect on the mixing action in both mixers. Secondly, differences in dough rheology were found. BF dough developed to maximum dough strength in a pin mixer resulted in a higher fracture stress (56.8 vs 50.0 kPa), a higher %recovery (66.6 vs. 64.1%) and a lower retardation time (39.2 vs 42.4s). This is in contrast with Mani et al. (1992) who concluded that fundamental rheological properties of optimally mixed doughs do not depend on the mixing equipment. The observed differences in dough rheology may be related to the different microstructure as observed by CSLM. The gluten strands of dough developed in the pin mixer appeared to be thinner and longer compared to the z-blade mixer resulting in higher resistance and elasticity. The different mixing action may explain the differences in microstructure. A pin mixer is generally viewed as mixing more 'uni-axially' resulting in thin extended gluten threads whereas a z-blade mixer also rotates and shears the dough more.

5.7. Conclusions

In this chapter the effect of mixing time, mixer type (pin mixer – z-blade mixer) and dough formulation (flour-water dough and baking formulation dough) was investigated on dough rheology and microstructure.

Upon prolonged mixing of FW dough, phase angle delta was shown to decrease. However, this was only observed at low frequency. At the same time also $J_{c,max}$ was found to decrease and elasticity of the dough increased, as measured by retardation times and %recovery. It is

thought that these results reflect the development and dispersion of the gluten network structure during mixing. Uniaxial extension properties were less sensitive to these changes. However, the decrease in extensibility and small increase in dough strength support the hypothesis.
The decrease in $J_{c,max}$ was shown to depend on mixer type, with a steeper decrease found for the pin mixer.

The incorporation of AA and salt to the FW dough, added both chemical and physical interactions to the dough system, thus increasing the complexity. It was the total response of these interactions which was measured. Addition of salt and ascorbic acid had a large effect on the rheological properties. Dynamic oscillation measurements showed to be sensitive to the addition of salt and AA to the dough formulation. However, they did not reflect changes in dough strength during mixing. In contrast, creep-recovery and uniaxial extension showed clear differences in dough strength occurring during dough development. A maximum dough strength was observed as a peak in R_{max}, Area and fracture stress, and as a minimum in $J_{c,max}$ and retardation times. At the same moment, a significant increase in %recovery and strain hardening index was observed. Both methods are thus useful in determining dough development to maximum strength.

CSLM revealed large differences in dough microstructure between the FW and BF doughs. The change in microstructure upon addition of salt and ascorbic acid offers an explanation for the changes in dough rheology. Furthermore, a different microstructure observed for BF dough developed in the pin mixer and the z-blade mixer, may explain the difference found in dough strength at optimal dough development.

Although a maximum dough strength was observed, bread quality was not negatively affected by mixing beyond maximum dough strength. This indicates that the properties as measured after mixing, do not entirely determine the final bread quality.
Between mixing and bread baking, a long breadmaking process is situated in which dough properties and microstructure may change. Therefore, the influence of the breadmaking process on dough rheology and microstructure is investigated in Chapter 6.

Chapter 6

Changes in dough rheology and microstructure during breadmaking

IFFRABOETRAMMEKEN: zeer dunne boterham.

[Oilsjtersen Diksjoneir]

6.1. Introduction

After dough formation during mixing, the dough is further processed before baking transforms the dough into bread. The next stages in bread manufacture are the subdivision of the bulk dough (dividing) and the shaping of individual dough pieces (moulding) to obtain the desired bread variety. Shaping is a multi-stage operation and may involve a further resting period between moulding stages (intermediate or first proof) (Marsh and Cauvain, 2007). Dough processing is an important factor determining the quality of bread. During processing steps, considerable changes in the structure and properties of the dough occur (Esselink et al., 2003). In this introduction, the description of the processing is limited to the production of standard pan bread. Other types of bread (e.g. baguettes) may involve different types of processing.

In most cases, dividing or scaling of the dough is followed by a rounding operation. By rounding the scaled pieces of dough are shaped into a round ball with smooth unbroken skin over its entire surface (Haegens, 2006). This will ensure gas retention and aid in the expansion of the dough piece. Usually the rounding operation is followed by an intermediate proof in which the dough is allowed to relax after undergoing a great deal of mechanical stress. The intermediate proof is often eliminated. This may lead to a reduction in loaf volume and a more open crumb texture due to damage to the bubble structure (Dobraszczyk, 2005). The action of rounding will add stresses and strains which may damage the existing dough structure. However, some breadmaking processes benefit from limited structural modification at this stage, especially when a longer intermediate proof is applied (Marsh and Cauvain, 2007).

Upon completion of the intermediate proof, the dough pieces are moulded into the desired shape prior to being placed into the baking pan. This operation involves three separate steps. First, the dough ball is transformed into a thin oval sheet during sheeting. In industrial practice this is performed by passing the dough piece through two or three sets of closely spaced rolls that progressively flatten and de-gas the dough (Dobraszczyk, 2005; Haegens, 2006). In addition to form the dough piece into an appropriate shape, dough moulding helps to sub-divide large gas bubbles within the dough and to generate an even distribution of bubbles throughout the dough piece. This ensures obtaining a bread with a fine and uniform crumb texture (Qi et al., 2008). Secondly, the sheeted dough piece is rolled into a cylindrical

form during the curling operation. Finally, the curled dough piece passes under a pressure board whose purpose is to eliminate any adventitious gas pockets within the dough and also to seal the seams of the loaf (Dobraszczyk, 2005; Haegens, 2006). Further steps are the panning of the dough, final proof and baking.

Optimal dough properties are required to obtain adequate processing and desired bread quality. The dough should not be too tight or slack (soft). A tight dough will offer greater resistance to deformation and is more prone to damage. The dough surface will tear and rupture leading to loss of quality. A soft dough will easily change its shape during moulding but it will lose its shape during proving and will start to flow. This is especially undesired for plate or hearth bread (Dobraszczyk, 2005). In sheeting, the shape of the dough changes and the rheological properties of the dough determine the stresses and strains. Repeated sheeting may be used to develop the gluten network in bread dough, which increases the elasticity of the dough (Dobraszczyk and Morgenstern, 2003). The sheeting of dough has an important role in determining the final quality of the dough (Autio and Laurikainen, 1997). Several researchers have tried to construct models for predicting dough behaviour during sheeting (Engmann et al., 2005; Mitsoulis and Hatzikiriakos, 2009; Qi et al., 2008).

6.2. Problem statement and research strategy

Although many publications exist on the effect of mixing on dough development and rheology, published research investigating changes in dough rheology and microstructure during breadmaking is limited. Dobraszczyk and Morgenstern (2003) also noticed that few studies have been done to find the relationship between fundamental rheological properties and sheeting.

In this chapter, the effect of breadmaking on dough rheology and microstructure was investigated. For this purpose doughs made from two wheat flours differing in quality, more specifically wheat varieties Bussard (good quality) and Tulsa (lower quality), were processed according to the standard breadmaking procedure and dough samples from six sampling points during breadmaking were analyzed. The dough formulation consisted of all ingredients except yeast. It is known that yeast has an important impact on dough rheology, but rheological measurements on yeasted dough are troubled by the gas production. The

exclusion of yeast from the bread dough formula, turned the fermentation periods in the baking procedure into resting periods.

Rheological investigation included oscillatory rheology, creep-recovery and uniaxial extension. Dough microstructure was visualized by CSLM. The aim of this research was thus to gain more insight in changes in dough rheology and microstructure occurring during dough processing.

Results will first be presented and will then be discussed with respect to the processing steps (rounding, moulding and resting phases) and for the comparison of Bussard and Tulsa flours.

6.3. Materials and methods

6.3.1. Wheat flour

Wheat flour of cultivar Bussard (good quality) was obtained from Paniflower (Ghent, Belgium). Wheat from cultivar Tulsa (lower quality, Clovis Matton) was tempered overnight to a moisture content of 15.5% and milled on the Bühler laboratory mill. An overview of the most important quality characteristics of the two wheat flours is given in Table 6.1.
Tulsa wheat flour shows low values for gluten index, Zeleny sedimentation value, dough development time, stability and alveograph deformation energy W. The large difference in protein content between Bussard and Tulsa flour, is probably the main responsible for this difference in quality parameters.

Table 6.1 Quality characteristics of Bussard and Tulsa wheat flour

	Bussard	*Tulsa*			*Bussard*	*Tulsa*
Protein content (%dm)	14.2	9.9	Farinograph*	WA (%)	58.0	56.3
Gluten index (%)	95.6	90.7		DDT (min)	5.5	2
Wet gluten (%)	34.3	23.0		Stability (min)	14.4	2.3
Zeleny (mL)	50	29	Alveograph**	P (mm H_20)	78	79
Ash content (%dm)	0.54	0.56		L (mm)	131	72
Damaged starch (%)	5.1	6.6		P/L	0.6	1.1
Falling number (s)	323	384		W (10^{-4} J)	322	181

*WA: water absorption; DDT: dough development time ** P: tenacity; L: extensibility; W: deformation energy

6.3.2. Dough preparation for rheological testing

As protein content has a large impact on dough rheology as discussed in Chapter 4, the two flours were standardized to a protein content of 11%dm. Native wheat starch (Cargill, Belgium) was added to the Bussard flour. To increase the protein content of the Tulsa flour, gluten was first isolated. Tulsa flour and deionised water were mixed in the farinograph to mixer peak consistency and hand washed to remove starch with deionised water to obtain the gluten fraction. The gluten was frozen, freeze-dried and milled. The freeze-dried Tulsa gluten had a protein content of 72.6% (on dm). The properties of the standardized wheat flours are shown in Table 6.2.

After standardization, the amount of protein and gluten in the wheat flours is comparable. However, the protein quality of Bussard flour is still better as shown by Zeleny and gluten index. Mixing properties are similar, however WA of Bussard is 2% lower than for Tulsa, probably caused by the higher amount of damaged starch present in the Tulsa flour. Rheological behaviour as measured by the alveograph is different for the two flours. The Bussard dough is softer and more extensible than the Tulsa dough.

Table 6.2 Properties of Bussard and Tulsa wheat flour after standardization to 11% protein

		Bussard	*Tulsa*
Gluten index (%)		96.1	91.8
Wet gluten (%)		26.6	26.5
Zeleny (mL)		37	33
Farinograph*	WA (%)	54.0	56.1
	DDT (min)	1.7	1.7
	Stability (min)	2.8	2.3
Alveograph**	P (mm H_20)	64	91
	L (mm)	126	72
	P/L	0.51	1.26
	W (10^{-4} J)	236	219

*WA: water absorption; DDT: dough development time
** P: tenacity; L: extensibility; W: deformation energy

Dough formulation for rheological testing consisted of the standardized wheat flour (Bussard or Tulsa), deionised water according to the farinograph water absorption (Bussard: 55.3%, Tulsa: 56.5%), 1.5% salt, 25 ppm ascorbic acid and malt flour (Bussard: 0.12%, Tulsa:

0.17%) to obtain a falling number of 250. Doughs were mixed in a 300 g farinograph mixer for 6 and 5 minutes for Bussard and Tulsa doughs respectively. After mixing and 10 min rest, dough was divided into 5 pieces (±85g) which were processed as in the standard breadmaking test (Figure 6.1). Dough samples for rheological testing were taken at six points during breadmaking: before and after rounding (S1-S2), before and after moulding (S3-S4) and halfway and at the end of the second 'fermentation phase' (S5-S6). As the bread formula did not contain yeast, the fermentation phases corresponded in fact to resting periods. Samples for rheometry (±15g) and uniaxial extension (±25g) were immediately frozen in liquid nitrogen and stored at -20°C. Dough preparation and sampling were repeated four times to obtain four independent replicates for every sampling point.

Breadmaking tests were performed with the standardized wheat flours according to 2.2.5.2.

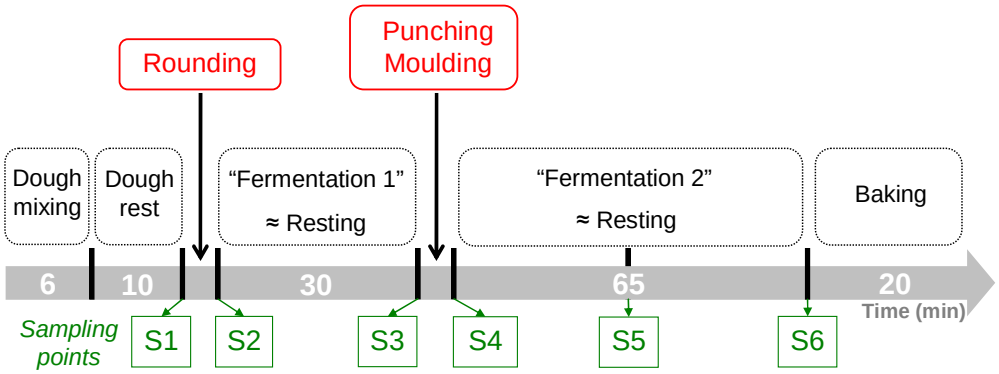

Figure 6.1 Breadmaking chart with dough sampling scheme

6.3.3. Dough rheology

Doughs were always analyzed the day after they were frozen. Doughs for rheological testing were defrosted prior to analysis by placing them in a closed container in a water bath at 25°C for 20 min.

For rheometry, the sample loading and relaxation procedure was the same as described in Chapter 3 (3.4.1). Prior to the frequency sweep measurements, the LVR of the Bussard and Tulsa doughs was determined for the different sampling points (S1-S6). Strain sweeps were performed at both 0.1 and 100 Hz on separate samples. Table 6.3 gives an overview of the

strains which indicate the end of the LVR. To be sure that all frequency sweeps are performed within the LVR, a strain value of 0.04% was chosen for all measurements.

Table 6.3. End of the linear viscoelastic region (%strain) for Bussard and Tulsa doughs as determined by strain sweeps at 0.1 and 100 Hz for doughs at different sampling points

	Bussard		*Tulsa*	
	0.1 Hz	*100 Hz*	*0.1 Hz*	*100 Hz*
S1	0.063	0.071	0.095	0.070
S2	0.047	0.063	0.075	0.070
S3	0.051	0.067	0.055	0.057
S4	0.053	0.065	0.074	0.066
S5	0.047	0.071	0.052	0.069
S6	0.055	0.070	0.046	0.064

After sample relaxation, a frequency sweep was performed in the frequency range 0.1 and 100 Hz. At the end of the frequency sweeps, a creep-recovery measurement was started with a creep phase of 5 min at a shear stress of 250 Pa. The creep phase was followed by a recovery phase of 10 min in which the shear stress was removed to allow the recovery of the sample.

Uniaxial extension measurements were performed as described in 2.2.4.3.

6.4. Results and discussion

6.4.1. Frequency sweeps

Dynamic oscillation frequency sweeps were applied to analyze the small deformation rheological properties. This allowed to evaluate the effect of the breadmaking procedure on dynamic oscillation parameters G', G", $|G^*|$ and phase angle δ. The results from the frequency sweeps performed on Bussard and Tulsa doughs sampled during breadmaking are shown in Table 6.4. It was chosen to summarize the frequency sweeps at two frequencies, more precisely at a low (0.1 Hz) and at a high (10 Hz) frequency.

Table 6.4 Dynamic moduli (G', G" and $|G^*|$) [Pa] and phase angle δ [°] of Bussard and Tulsa dough for different sampling points (S1-S6) during breadmaking [*]

	0.1 Hz				*10 Hz*			
	G'	*G"*	$\|G^*\|$	*δ*	*G'*	*G"*	$\|G^*\|$	*δ*
Bussard								
S1	7069 [a]	2112 [a]	7378 [a]	16.6 [a]	15940 [a]	6241 [a]	17117 [a]	21.4 [a]
S2	7814 [a]	2360 [a]	8162 [a]	16.8 [a]	17450 [a]	6837 [a]	18740 [a]	21.4 [a]
S3	7221 [a]	2131 [a]	7529 [a]	16.4 [a]	16110 [a]	6322 [a]	17310 [a]	21.4 [a]
S4	9202 [a]	2691 [a]	9588 [a]	16.3 [a]	20130 [a]	7777 [a]	21580 [a]	21.2 [a]
S5	7852 [a]	2321 [a]	8188 [a]	16.5 [a]	17553 [a]	6896 [a]	18860 [a]	21.5 [a]
S6	8395 [a]	2589 [a]	8786 [a]	16.9 [a]	18820 [a]	7464 [a]	20247 [a]	21.7 [a]
Tulsa								
S1	7232 [a]	2263 [a,b]	7578 [a]	17.1 [a]	16083 [a]	6142 [a]	17373 [a]	20.9 [a,b]
S2	7939 [a,b]	2407 [a,b]	8296 [a,b]	16.8 [a,b]	16685 [a]	6332 [a]	18845 [a,b]	20.8 [a]
S3	7184 [a]	2172 [a]	7505 [a]	16.8 [a,b]	16388 [a]	6220 [a]	17038 [a]	20.8 [a]
S4	8664 [b]	2576 [b]	9039 [b]	16.6 [b]	19083 [a]	7210 [a]	20398 [b]	20.7 [a]
S5	8229 [a,b]	2526 [a,b]	8607 [a,b]	17.1 [a,b]	18393 [a]	7070 [a]	19705 [a,b]	21.0 [a,b]
S6	7585 [a,b]	2401 [a,b]	7956 [a,b]	17.6 [c]	17238 [a]	6698 [a]	18603 [a,b]	21.2 [b]

[*] Values indicated with different letters in the same column are significantly different (α=0.05)

For both dough systems similar trends are observed in G' and G" during the breadmaking procedure. As a result of rounding (S2) and moulding (S4) G' and G" show an increase. During the following resting periods values for G' and G" decrease again. However, no significant changes could be detected for the Bussard dough. Also for phase angle δ no significant changes could be observed. The lowest value for δ was found after the moulding step. For the Tulsa dough, significant changes were observed. G' (0.1 Hz), G" (0.1Hz) and $|G^*|$ (0.1 and 10 Hz) increase significantly due to the moulding step (S4). A minimum in phase angle δ occurs at the same time. During the subsequent resting phase, the dynamic moduli decrease and the phase angle increases again indicating dough relaxation.

In Figure 6.2 the small deformation rheological properties of Bussard and Tulsa doughs obtained at the beginning (S1) and at the end (S6) of the breadmaking procedure are compared. The curves for G' and phase angle δ show an upward trend with increasing frequency as already observed in Chapter 5. After mixing (S1) both doughs have comparable values for G'. At the end of the breadmaking process (S6) values for G' are slightly higher for both doughs with the highest value obtained for Bussard dough, however differences were not significant.

Similar to G', also phase angle δ didn't differ much between Bussard and Tulsa dough and the breadmaking process did not cause major changes in δ. However, a significant difference was found at high frequency (100Hz) between the doughs at the end of breadmaking (S6). Bussard dough shows a higher phase angle indicating more fluid character.

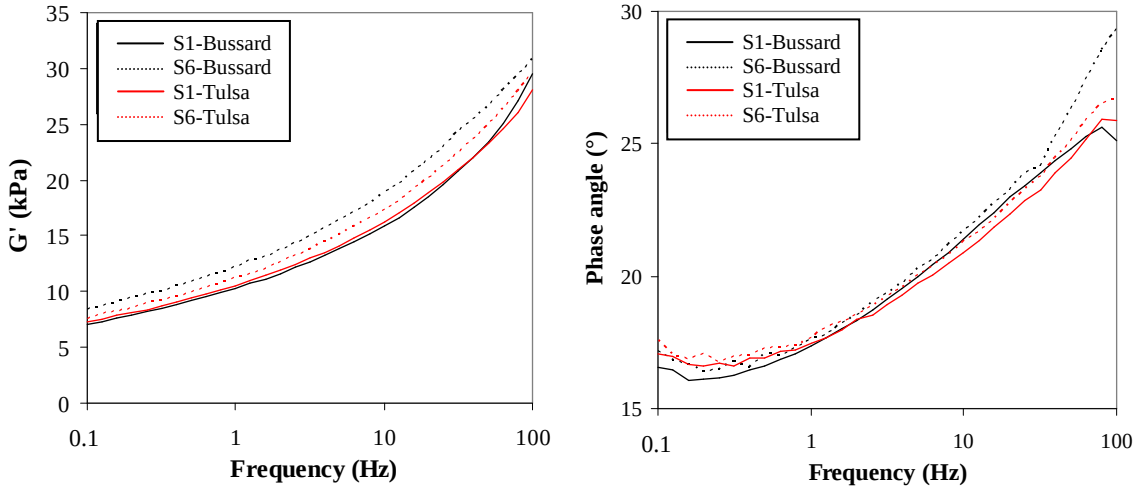

Figure 6.2 Comparison between Bussard and Tulsa dough for the elastic modulus G' and the phase angle δ at the beginning and the end of the breadmaking process (S1 and S6 respectively)

6.4.2. Creep-recovery

The effect of breadmaking on the maximum creep compliance $J_{c,max}$ is shown in Figure 6.3 for Bussard and Tulsa doughs. It can be seen that Bussard and Tulsa doughs show a similar deformation behaviour under the applied shear stress of 250 Pa.

The breadmaking procedure causes changes in $J_{c,max}$ and the changes are observed for both wheat cultivars. $J_{c,max}$ is not significantly changed by rounding (S2). The moulding step (S4), on the other hand, causes a significant decrease. During the resting phases (S3-S5-S6), $J_{c,max}$ increases again, due to sample relaxation.

Although $J_{c,max}$ is influenced by dough processing steps, dough at the end of the process (S6) shows a similar or only a slightly higher $J_{c,max}$ as after mixing (S1).

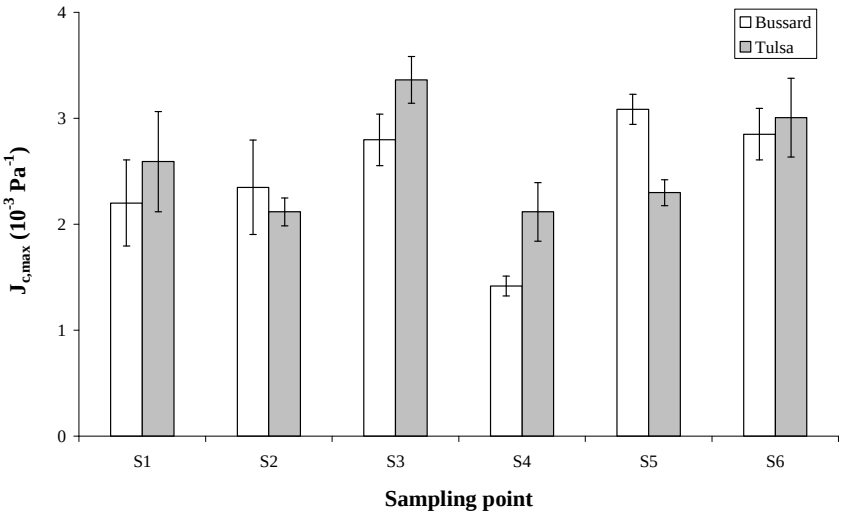

Figure 6.3 Maximum creep compliance ($J_{c,max}$) during breadmaking for Bussard and Tulsa doughs

The recovery characteristics of the doughs are listed in Table 6.5. When looking at the effect of the different steps during breadmaking it is found that the rounding step (S2) does not influence the recovery properties of the dough. On the other hand, moulding (S4) results in doughs with higher strength and elasticity which is seen as an increase in %recovery (Tulsa: 44.7% before vs 56.1% after moulding) and lower retardation times (r_1 for Bussard and r_2 for Tulsa). When the dough relaxes in the following fermentation phase, the dough loses part of its elastic strength as %recovery decreases (Bussard: 60.6% (S4) and 52.5% (S6)). Bussard dough shows stable values for retardation time r_2 whereas significant changes are observed for Tulsa dough. This also indicates the better stability of Bussard dough during the process.

Table 6.5 Recovery properties of Bussard and Tulsa doughs sampled during breadmaking (S1-S6): total recovery compliance $J_{r,max}$, percentage recovery ($J_{r,max}/J_{c,max}$*100) and retardation times r_1 and r_2 [*]

	Bussard dough				*Tulsa dough*			
	$J_{r,max}$ (10^{-3} Pa^{-1})	%recovery (-)	r_1 (s)	r_2 (s)	$J_{r,max}$ (10^{-3} Pa^{-1})	%recovery (-)	r_1 (s)	r_2 (s)
S1	1.22 ± 0.12 ^{a}	56.0 ± 4.8 ^{a,b,c}	1.0 ± 0.1 ^{a}	35.1 ± 1.5 ^{a}	1.30 ± 0.14 $^{a.b}$	50.8 ± 4.0 $^{a.c}$	1.1 ± 0.1 $^{a.b}$	39.9 ± 1.4 $^{a.c}$
S2	1.25 ± 0.17 ^{a}	53.6 ± 4.4 ^{a,b,c}	1.0 ± 0.1 ^{a}	34.9 ± 0.5 ^{a}	1.15 ± 0.04 ^{a}	54.4 ± 2.1 $^{a.c}$	1.1 ± 0.1 $^{a.b}$	38.6 ± 0.6 $^{a.b}$
S3	1.55 ± 0.09 ^{a}	55.6 ± 4.6 ^{a,b,c}	1.0 ± 0.1 ^{a}	35.4 ± 0.2 ^{a}	1.50 ± 0.08 ^{b}	44.7 ± 1.1 ^{b}	1.1 ± 0.1 $^{a.b}$	39.2 ± 0.4 $^{a.c}$
S4	0.86 ± 0.04 ^{b}	60.6 ± 1.5 ^{a}	0.8 ± 0.1 ^{b}	34.3 ± 0.5 ^{a}	1.18 ± 0.13 ^{a}	56.1 ± 2.0 ^{c}	1.1 ± 0.1 ^{a}	37.4 ± 0.6 ^{b}
S5	1.49 ± 0.07 ^{a}	48.5 ± 0.1 ^{b}	1.0 ± 0.1 ^{a}	35.6 ± 0.4 ^{a}	1.23 ± 0.05 $^{a.b}$	53.5 ± 1.0 $^{a.c}$	1.1 ± 0.1 $^{a.b}$	38.7 ± 0.6 $^{a.b}$
S6	1.50 ± 0.12 ^{a}	52.5 ± 0.5 ^{c}	1.0 ± 0.1 ^{a}	36.2 ± 0.6 ^{a}	1.47 ± 0.16 ^{b}	49.0 ± 1.3 $^{a.b}$	1.2 ± 0.1 ^{b}	40.9 ± 0.4 ^{c}

[*] Values indicated with different letters in the same column are significantly different (α=0.05)

Although changes occurred in recovery properties during the breadmaking steps, no differences were observed between the doughs at the beginning and at the end of breadmaking (S1 and S6).

When comparing Bussard and Tulsa dough at the end of breadmaking (S6), their creep deformation behaviour was similar. On the other hand, differences in recovery characteristics were observed. Compared to Tulsa dough, Bussard dough shows a significantly higher %recovery (52.5% vs. 49.0%) and a lower retardation time r_2 (36.2s vs 40.9s), indicating more elastic properties.

6.4.3. Uniaxial extension properties

Next to dynamic oscillation (small shear deformation) and creep-recovery (larger shear deformation) also uniaxial extension tests (large extension deformation) were performed. Uniaxial extension properties of the Bussard and Tulsa doughs sampled during the breadmaking procedure are listed in Table 6.6. R_{max}, Ext and Area of Bussard dough remained stable during the breadmaking procedure. Only an increase in Area was observed at the end of the last resting phase (S6). This means that at that point, Bussard dough requires more energy for extension until rupture.

For Tulsa dough, an increase in R_{max} was found during breadmaking. However, R_{max} decreased again during the second resting phase (S5-S6). Rounding (S2) caused a significant decrease in Ext which remains stable upon further processing. Also, Area decreased significantly at the end of breadmaking (S5-6).

Table 6.6 Uniaxial extension properties for Bussard and Tulsa doughs sampled during breadmaking*

	Bussard dough			*Tulsa dough*		
	R_{max} (N)	Ext (mm)	Area (N.mm)	R_{max} (N)	Ext (mm)	Area (N.mm)
S1	0.33 ± 0.07 ^{a}	52.2 ± 6.2 ^{a}	11.2 ± 1.2 ^{a,b}	0.23 ± 0.02 ^{a}	63.3 ± 4.5 ^{a}	10.4 ± 0.8 ^{a}
S2	0.31 ± 0.05 ^{a}	53.2 ± 5.6 ^{a}	11.4 ± 1.3 ^{a,b}	0.30 ± 0.04 ^{b}	48.3 ± 5.2 ^{b}	9.9 ± 1.1 ^{a,b}
S3	0.30 ± 0.03 ^{a}	50.7 ± 3.8 ^{a}	10.3 ± 1.1 ^{a}	0.31 ± 0.04 ^{b}	47.8 ± 4.3 ^{b}	10.0 ± 1.0 ^{a,b}
S4	0.34 ± 0.06 ^{a}	48.7 ± 4.8 ^{a}	10.7 ± 1.3 ^{a}	0.31 ± 0.03 ^{b}	45.8 ± 2.6 ^{b}	9.9 ± 0.9 ^{a,b}
S5	0.27 ± 0.06 ^{a}	56.2 ± 5.8 ^{a}	10.2 ± 1.1 ^{a}	0.22 ± 0.03 ^{a}	51.0 ± 3.6 ^{b}	7.8 ± 1.1 ^{c}
S6	0.34 ± 0.05 ^{a}	55.2 ± 6.1 ^{a}	12.6 ± 0.8 ^{b}	0.26 ± 0.03 ^{a,b}	46.7 ± 3.2 ^{b}	8.5 ± 1.2 ^{b,c}

*Values indicated with different letters in the same column are significantly different (α=0.05)

From the uniaxial extension measurements, the dough fracture properties were calculated (2.2.4.3) of which the results are shown in Table 6.7. For Bussard dough fracture stress (σ_{max}), fracture strain (ε_H) and strain hardening index (SHI) were constant throughout breadmaking except for an increase in fracture stress which was observed at the end of breadmaking (S6). This corresponds with the observed increase in Area (Table 6.6).

Fracture stress of Tulsa dough significantly decreased in the last resting phase (S5-6). Further, rounding (S2) causes a significant decrease of the fracture strain which corresponds with the reduced extensibility. For Tulsa dough also a maximum SHI was found after the moulding step (S4).

Table 6.7 Dough fracture properties calculated from uniaxial extension for Bussard and Tulsa doughs sampled during breadmaking*

	Bussard dough			*Tulsa dough*		
	σ_{max} (kPa)	ε_H (-)	SHI (-)	σ_{max} (kPa)	ε_H (-)	SHI (-)
S1	51.5 ± 6.6 ^{a,b}	1.8 ± 0.1 ^{a}	1.74 ± 0.08 ^{a}	47.7 ± 4.2 ^{a}	2.1 ± 0.1 ^{a}	1.63 ± 0.04 ^{a}
S2	48.8 ± 4.4 ^{a}	1.8 ± 0.1 ^{a}	1.69 ± 0.07 ^{a}	44.1 ± 5.4 ^{a,b,c}	1.8 ± 0.1 ^{b}	1.73 ± 0.08 ^{a,b}
S3	45.9 ± 4.7 ^{a}	1.8 ± 0.1 ^{a}	1.72 ± 0.05 ^{a}	46.0 ± 4.4 ^{a,c}	1.8 ± 0.1 ^{b}	1.67 ± 0.06 ^{a,b}
S4	49.8 ± 6.2 ^{a}	1.8 ± 0.1 ^{a}	1.80 ± 0.11 ^{a}	43.0 ± 4.6 ^{a,b,c}	1.8 ± 0.1 ^{b}	1.76 ± 0.06 ^{b}
S5	47.1 ± 4.4 ^{a}	1.9 ± 0.1 ^{a}	1.72 ± 0.05 ^{a}	36.0 ± 5.4 ^{b}	1.8 ± 0.1 ^{b}	1.65 ± 0.05 ^{a}
S6	58.4 ± 1.8 ^{b}	1.9 ± 0.1 ^{a}	1.68 ± 0.08 ^{a}	38.8 ± 5.6 ^{b,c}	1.7 ± 0.1 ^{b}	1.68 ± 0.04 ^{a,b}

*Values indicated with different letters in the same column are significantly different (α=0.05)

6.4.4. Microstructure

Microstructure of Bussard and Tulsa dough sampled during breadmaking was visualised by CSLM. The images for the six sampling points [S1-6] during breadmaking are shown in Figure 6.4 for Bussard dough and Figure 6.5 for Tulsa dough.

For the Bussard doughs, a better structured protein network is observed when the dough was rounded (S2). It seems that rounding structures the gluten network which is seen as more visible and thicker gluten strands compared to the dough before rounding (S1). Similar structures can be seen at the end of the first resting period (S3). The moulding step (S4) has a clear effect on dough microstructure. Gluten strands are finer, shorter and more interconnected. This structure remains present during the second resting phase (S5-6).

In contrast to the Bussard dough, rounding (S2) caused no major changes of Tulsa dough microstructure. Dough after the moulding step (S4) shows a dense network of very fine gluten strands which are aligned in the same direction. The gluten strands seem to be finer and less interconnected compared to the Bussard dough. The fine gluten structure remains present during the last resting period (S5-6).

6.4.5. Breadmaking tests

Breadmaking tests were performed with the standardised wheat flours of Bussard and Tulsa. The results are summarised in Table 6.8. The Bussard flour clearly has a better quality as it showed a significantly higher oven rise and bread volume for the pan breads. Although the height of the Tulsa dough at the end of the second fermentation phase was higher, the oven rise was limited which resulted in a lower bread volume.

For the plate breads, no significant difference was found for the bread volume but a higher form ratio H/W was found for the Bussard breads indicating that Bussard dough exhibited more elastic properties. Plate breads give information about the ability of the dough to resist a flow which is important to retain a proper shape in plate bread production (Faergestad et al., 2000).

Table 6.8 Breadmaking quality of Bussard and Tulsa wheat flour standardised to 11%dm protein[*]

	Bussard	Tulsa
Pan bread		
Dough height (mm)[**]	71.8 ± 0.9 [a]	74.5 ± 0.7 [b]
Oven rise (%)	11.0 ± 0.8 [a]	7.5 ± 0.9 [b]
Bread mass (g)	102.8 ± 0.4 [a]	102.4 ± 0.4 [a]
Bread volume (cm^3/100g flour)	451 ± 6 [a]	438 ± 4 [b]
Plate bread		
Bread mass (g)	81.8 ± 0.3 [a]	80.9 ± 0.3 [b]
Bread volume (cm^3/100g flour)	492 ± 21 [a]	463 ± 11 [a]
H/W	0.76 ± 0.01 [a]	0.68 ± 0.01 [b]

[*]Values indicated with different letters in the same row are significantly different (α=0.05)
[**]Dough height before baking

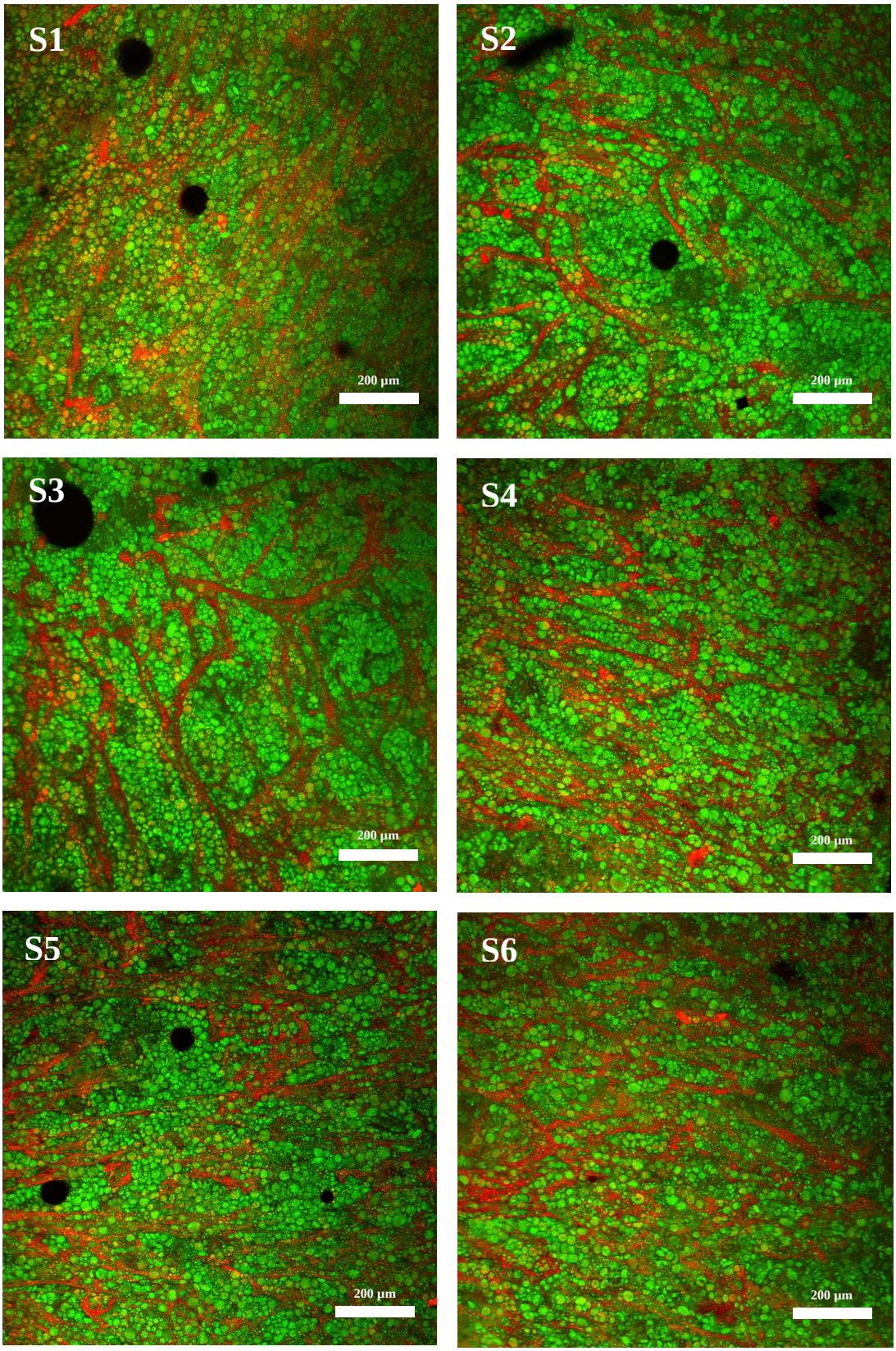

Figure 6.4. CSLM images of Bussard dough during the breadmaking process (green=starch granules, red=protein network, bar=200 µm) [S1-2: before and after rounding; S3-4: before and after moulding; S5-6: halfway and end of second 'fermentation' period]

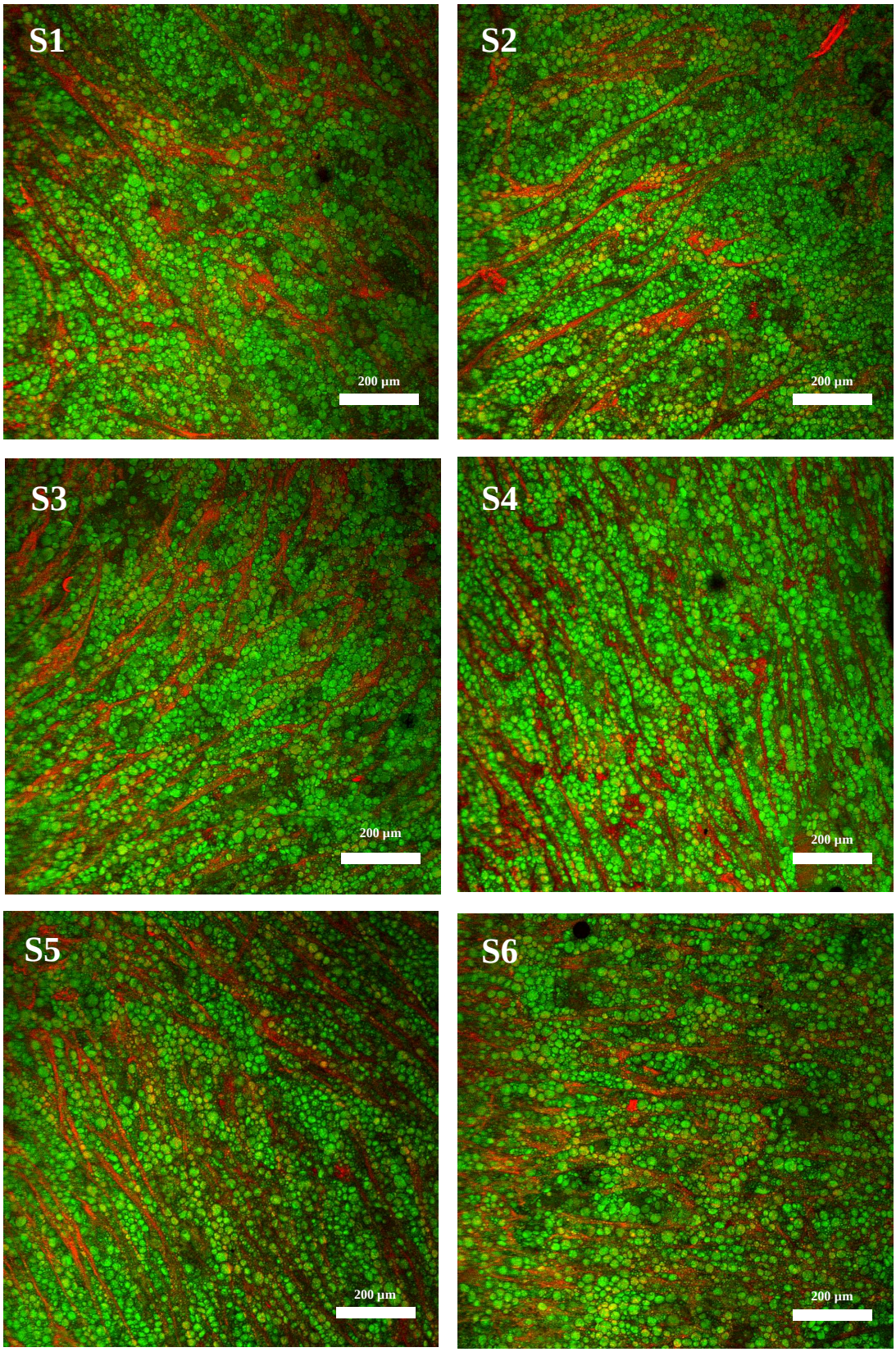

Figure 6.5. CSLM images of Tulsa dough during the breadmaking process (green=starch granules, red=protein network, bar=200 µm) [S1-2: before and after rounding; S3-4: before and after moulding; S5-6: halfway and end of second 'fermentation' period]

6.5. Discussion

6.5.1. Impact of the breadmaking process on dough rheology and microstructure

6.5.1.1. Rounding

Rounding was the first processing step of interest in the followed breadmaking procedure. During rounding, a piece of dough is shaped in such a way that a symmetric dough ball is obtained with a sufficiently thick and continuous skin that will retain the freshly produced gas and thus aid the expansion of the dough piece during the intermediate proof (Dobraszczyk, 2005). In this study, the rounding step did not cause major changes in dough rheological properties as measured by small and large deformation tests. The only significant change in dough properties was observed for Tulsa dough which seemed to be strengthened as R_{max} increased and Ext decreased. However, no visible changes in Tulsa dough microstructure were observed (Figure 6.5). On the other hand, Bussard dough seemed to contain a more structured gluten network after rounding.

6.5.1.2. Moulding

During the moulding operation, dough is sheeted and curled to obtain the dough rolls which are placed in the baking pans. In contrast to rounding, moulding caused significant changes in dough rheology as observed by dynamic oscillation and creep-recovery. Surprisingly, uniaxial extension properties were not significantly affected by moulding. Doughs after moulding showed higher G', G", $|G^*|$ and %recovery, and lower $J_{c,max}$ and retardation times. These results indicate changes in the gluten network structure leading to an enhanced dough strength and elasticity. It is known that sheeting helps to further develop the gluten network resulting in higher elasticity of the dough (Dobraszczyk and Morgenstern, 2003). Esselink et al. (2003) observed an increase in stress during dough compression test as a result of the sheeting operation.

The changes in dough rheology may be related to the changes in microstructure as observed in Figure 6.4 and Figure 6.5. The high forces exerted on the dough system during moulding, cause a structural reorganisation of the gluten matrix. For Bussard doughs this results in a very strong interconnected gluten network. For Tulsa dough, a network of fine gluten strands is obtained which are less interconnected. This difference in network structure may explain

the much higher $J_{c,max}$ found for Tulsa after moulding. At this point Bussard doughs also showed a higher %recovery and lower retardation times compared to the Tulsa dough which is an indication of a more elastic dough system. Microstructural studies have shown that sheeting results in the organization of the protein network in the dough and that excessive sheeting can break it down (Autio et al., 1997). Cryo-SEM images have demonstrated that sheeting may lead to strong orientation and sometimes disruption of the gluten strands (Esselink et al., 2003).

6.5.1.3. Resting periods

The fermentation phases following the processing steps are in fact resting periods in which the dough is allowed to relax from the imposed deformation. This is seen in the evolution of the dough rheological parameters as G', G", $|G^*|$ and %recovery decrease and $J_{c,max}$, phase angle δ and the retardation times increase again. This is in contrast with Kim et al. (2008) who observed no effect or an increase in $|G^*|$ upon resting for optimally mixed dough from a weak and a strong flour respectively. It has been shown that during resting, glutenin particles which have been homogeneously distributed among the dough, re-assemble. For longer resting times this is accompanied by an increase in relaxation half times which is an indication for an increase in elasticity of the dough (Don et al., 2005). Changes in gluten structure may be a possible explanation for the strengthening of Bussard dough and the weakening of Tulsa dough during the last resting phase as observed through uniaxial extension tests. However, the dough samples contained the whole bread formula except yeast, where Don et al. (2005) and Kim et al. (2008) studied simple flour-water doughs.

No changes in microstructure were observed during the resting phases (Figure 6.4 and Figure 6.5). So the observed differences in rheology may result from the relaxation phenomena in the gluten structure occurring in the dough during resting.

Although changes in dough microstructure were observed by CSLM, no large differences in dough rheology were found between doughs after mixing (S1) and at the end of the breadmaking procedure (S6). Only Tulsa seemed to have lost some dough strength as Area and σ_{max} at S6 were significantly lower than at S1.

6.5.2. Bussard vs. Tulsa

In this study, two flour types of different quality were used. To overcome the effect of protein content, the flours were standardised to a protein content of 11%. Next to protein content also wet gluten content was shown to be equal for the two standardised flours. However, the gluten index and Zeleny sedimentation value indicated the Bussard flour to be the better quality flour. This was also shown in the breadmaking test as a significantly higher oven rise and bread volume were found for the Bussard flour.

After mixing, both doughs showed similar properties for dynamic oscillation and creep-recovery parameters. A slightly higher phase angle delta was observed for Tulsa at 0.1 Hz (17.1 vs. 16.6°), but at higher frequency, Bussard dough showed the highest phase angle (21.4 vs. 20.9°). Further, differences were observed in uniaxial extension parameters as Tulsa dough showed lower R_{max} and larger Ext. The results from the alveograph already indicated a large difference in P/L between the two standardized flours (Bussard: 0.51 vs Tulsa: 1.26). When applying the model (protein content, WA and P/L) which was shown to best predict bread volume in Chapter 4, volumes of 455 and 442 $cm^3/100g$ flour are predicted for standardized Bussard and Tulsa flours. This closely resembles the results from the breadmaking tests. However, taking into account the error on the predicted value, bread volumes are not significantly different.

During processing, a difference in behaviour between the two doughs was observed. Bussard dough was found to be more resistant to processing. No large changes occurred for phase angle δ, retardation times or uniaxial extension parameters. Whereas Tulsa was significantly affected by rounding (reduced Ext and increased R_{max}) and large changes were observed during the resting periods for phase angle δ, creep-recovery and uniaxial extension. This indicates that the viscoelastic structure of the Tulsa dough was less stable.

CSLM images support this conclusion as a more interconnected gluten network was found for the Bussard dough and the Tulsa dough showed a network of very fine threads with less interactions. The difference in microstructure may also explain the different creep-recovery behaviour as the Bussard dough clearly behaves more resistant and elastic than Tulsa dough after moulding. However, during subsequent resting, differences in creep-recovery behaviour disappear again.

The difference in microstructure may also explain the different baking results. Before baking, the height of the Bussard doughs were significantly lower compared to the Tulsa doughs. It is thought that this was caused by the stronger gluten network of the Bussard dough as observed by CSLM. This stronger network possibly restricted expansion of the dough during fermentation. However, this stronger network is able to support the dough expansion during oven rise resulting in a higher bread volume (451 vs 438 cm^3/100g flour) compared to Tulsa. Differences in baking behaviour are also shown by the difference in form ratio of the plate breads. Bussard (0.76) shows a significantly higher form ratio than Tulsa (0.68), indicating the superior elastic properties of the Bussard dough.

6.6. Conclusions

In this chapter the goal was to gain more insight in the changes occurring in dough rheology and dough microstructure during dough processing. For this purpose, dough of two wheat flours differing in quality, Bussard (good quality) and Tulsa (lower quality), was processed according to the breadmaking procedure and dough samples taken during breadmaking were analyzed for their rheological properties and microstructure.

By means of CSLM it was possible to elucidate clear differences in microstructure between Bussard and Tulsa dough. Gluten network structures were obviously affected by rounding and moulding resulting into a different microstructure, with a more interconnected gluten network for Bussard dough. In addition, creep-recovery tests showed clearly that Bussard dough was more resistant to deformation after the moulding step.

Processing had a significant effect on dough rheology as determined by dynamic oscillation and creep-recovery. Uniaxial extension properties seemed to be less harmed by processing as changes in those parameters were limited. This suggests that rotational rheometry is a suitable technique for monitoring changes in rheological properties due to processing.

Although the two wheat flours used in this study did not have significantly different properties after mixing, a clear difference in baking quality was observed. Changes in microstructure and rheology induced by dough processing may offer an explanation for the differences in baking results. These results indicate the difficulty of relating dough rheological properties to final baking quality.

In this study, yeast was not included in the bread formula because of the obstructive effect of yeast fermentation on the rheological measurements. It may be expected that the rheological behaviour of fermented doughs will be different due to metabolite production during fermentation. Further, the presence of gas bubbles in the dough will change the impact of processing on dough properties. Elucidating the role of fermentation on dough rheology and microstructure, presents new challenges.

General Conclusions

The rheological properties of a bread dough play an important role in dough processing and influence bread quality. In this research rotational rheometry comprising of dynamic oscillation and creep-recovery was applied to study dough rheology. The aim was to gain more insight in changes in dough (micro)structure as a result of variations in wheat flour quality, mixing and breadmaking.

Summary of major findings

First the methodology for applying rotational rheometry for analyzing dough rheology was optimized. A careful standardization of the sample preparation and the sample loading procedure made it possible to obtain reproducible results for dough which is highly sensitive for manipulation. Dynamic oscillation tests are a useful tool for monitoring normal force decay after sample loading and to determine the linear viscoelastic properties of a dough system. Creep-recovery is an interesting technique for evaluation of the non-linear viscoelastic properties of wheat flour dough. By applying the Burgers model, the elastic, retarded elastic and viscous components of a dough system can be described. In this research, the six parameter Burgers model was found to describe best the creep-recovery data. From the obtained parameters, only the retardation times, which are a measure of how fast recovery occurs after a controlled deformation, contains relevant information on dough elasticity. However, retardation times are highly sensitive to the applied creep-recovery methodology.

When analyzing the flour-water doughs of 17 pure wheat cultivars of varying quality, large differences in dough viscoelastic properties were observed by rotational rheometry although the doughs were prepared to similar consistency as measured by the farinograph. This suggests that applying farinograph water absorption does not result in dough systems similar

in consistency. Rotational rheometry is thus found to be a better measure concerning the overall consistency of a flour-water dough system.

With respect to wheat flour quality control, it was found that the rheological properties of the flour-water doughs obtained from rotational rheology were related to the breadmaking potential of the wheat flours, as measured by bread volume. In general, it can be concluded that when the flour-water dough shows a higher flow behaviour (higher phase angle, lower moduli, higher creep and recovery deformation), the dough will show a larger expansion ability in breadmaking (larger bread volumes). However, this does not give information on the ability of the dough matrix to stabilize the gas cell membranes and thus prevent premature rupture. In general, it can be stated that the small deformation properties give information about the effective water distribution in the dough and the interactions existing between the different dough components.

Principal component analysis on the creep-recovery parameters obtained from the Burgers model was able to group wheat cultivars with similar rheological properties. This grouping was based on the overall deformability during creep-recovery and retardation time r_2. This again indicates that retardation time is a better parameter for describing dough elasticity than recovery compliance.

It has been shown that rheological properties determined by small and large deformation in shear are strongly related. For flour-water doughs, a power law relationship was found between phase angle δ, reflecting dough microstructure, and the deformation occurring under creep. This relation was observed for dough samples obtained from different wheat cultivars as well as for dough samples from one bread wheat flour obtained at different stages during mixing. This suggests that dough microstructure may determine dough behaviour under larger shear deformation.

The mixing process was found to significantly affect dough rheology and microstructure of flour-water dough. Changes in dough strength and elasticity could be measured by rotational rheometry. Phase angle delta, maximum creep compliance, %recovery and retardation times all reflected changes in dough structure during mixing. They may thus be used to determine optimum dough development. Mixer type was found to influence dough development.

Addition of salt and ascorbic acid had a large effect on dough rheology and microstructure. For the baking dough formulation, dynamic oscillation was not sensitive to changes in dough strength during mixing. Creep-recovery and uniaxial extension on the other hand, could be used to indicate maximum dough strength. Differences were observed between doughs mixed in the pin and z-blade mixer, with a stronger and more elastic dough being formed in the pin mixer. This could be related to the observed changes in microstructure by CSLM which may result from the different mixing action.

As a large change in dough rheology and microstructure was observed between a flour-water dough and the baking formulation dough, it is recommended to resemble bread dough composition and preparation as closely as possible when investigating wheat flour functionality for breadmaking in further research.

CSLM revealed changes in dough microstructure caused by breadmaking for two wheat flours differing in protein quality. Especially the sheeting step caused major changes in the gluten network structure which could explain the difference in bread volume. Only creep-recovery was found to be sensitive to the changes in microstructure due to sheeting. Although the two tested wheat flours did not differ much in their rheological properties after mixing, their microstructure was differently affected by breadmaking. This may explain the difficulty of linking dough rheological parameters to breadmaking functionality of wheat flour.

Recommendations

In this research the methodology for applying rotational rheometry to analyze dough viscoelasticity was optimized. The method has been successfully applied to monitor changes in dough rheology and microstructure due to changes in flour quality, mixing and dough processing. Relating fundamental rheology to wheat flour functionality and the quality of bakery products has been and still remains a challenge for many researchers. In this research, a relation between fundamental rheology performed in shear and bread volume was found. This is in contrast with the general held view that measurements in shear do not at all relate to breadmaking potential. However, more research is needed to investigate whether rotational rheometry is applicable in wheat flour quality control and is able to differentiate among wheat flours which do not vary much in protein content and water absorption, wheat flours used in

industry or wheat flours obtained from other geographical regions. However, the baking test will remain the true test for evaluating the functionality of a wheat flour in breadmaking.
The major drawbacks for using this methodology in quality control are the investment cost, the need of skilled staff , the need for a thorough standardization of the methodology and the fact that it is time consuming. On the other hand, this research has proven that rotational rheometry is a sensitive tool for detecting changes in microstructure and that microstructure also influences final product quality. Small deformation properties were shown to give information about the effective water distribution in the dough and the interactions existing between the different dough components. Thus, the methodology may be a useful tool for investigating ingredient functionality and effects of processing on dough viscoelasticity and microstructure. However, next to rotational rheometry, it is still recommended to perform large extensional measurements (uniaxial or biaxial) as well because this type of deformation is predominant during fermentation and baking. It would be interesting to further investigate the relation between dough microstructure and extensional behaviour of bread dough.

In this research, CSLM proved to be a valuable tool for visualization of the dough microstructure. By using CSLM images it was possible to explain the changes in rheology caused by dough formulation, mixing and dough processing. The major disadvantages of this method are the sample preparation which may cause changes to the microstructure by manipulation and the limitation to 2D images. Further research could focus on other visualization tools such as x-ray tomography for monitoring bubble growth and bubble stability in bread dough and relate these findings to dough rheology and microstructure. Getting more insight in the actual deformations occurring during processing is necessary to further elucidate the relation between dough rheology and product quality.

References

AACC (1990). Standard methods of the American Association of Cereal Chemists. AACC: St. Paul.

Abdelrahman, A., and Spies, R. (1986). Dynamic rheological studies of dough systems. In "Fundamentals of dough rheology" (H. Faridi and J. M. Faubion, eds.), pp. 87-103. American Association of Cereal Chemists, St. Paul.

Aibara, S., Ogawa, N., and Hirose, M. (2005). Microstructures of bread dough and the effects of shortening on frozen dough. Bioscience Biotechnology and Biochemistry 69, 397-402.

Amend, T., and Belitz, H. D. (1990). The formation of dough and gluten - A study by scanning electron-microscopy. Zeitschrift Fur Lebensmittel-Untersuchung Und -Forschung 190, 401-409.

Anderssen, R. S., Gras, P. W., and MacRitchie, F. (1998). The rate-independence of the mixing of wheat flour dough to peak dough development. Journal of Cereal Science 27, 167-177.

Anjum, F. M., Khan, M. R., Din, A., Saeed, M., Pasha, I., and Arshad, M. U. (2007). Wheat gluten: High molecular weight glutenin subunits - Structure, genetics, and relation to dough elasticity. Journal of Food Science 72, R56-63.

Autio, K., Flander, L., Kinnunen, A., and Heinonen, R. (2001). Bread quality relationship with rheological measurements of wheat flour dough. Cereal Chemistry 78, 654-657.

Autio, K., and Laurikainen, T. (1997). Relationships between flour/dough microstructure and dough handling and baking properties. Trends in Food Science & Technology 8, 181-185.

Baltsavias, A., Jurgens, A., and van Vliet, T. (1997). Rheological properties of short doughs at small deformation. Journal of Cereal Science 26, 289-300.

Bauer, N., Koehler, P., Wieser, H., and Schieberle, P. (2003). Studies on effects of microbial transglutaminase on gluten proteins of wheat. II. Rheological properties. Cereal Chemistry 80, 787-790.

Bechtel, D. B., Abecassis, J., Shewry, P. R., and Evers, A. D. (2009). Development, structure and mechanical properties of the wheat grain. In "Wheat Chemistry and Technology" (K. Khan and P. R. Shewry, eds.), pp. 51-95. AACC International, St. Paul.

Belton, P. S. (1999). On the elasticity of wheat gluten. Journal of Cereal Science 29, 103-107.

Belton, P. S., and Dobraszczyk, B. J. (2006). Letter to the Editor. Journal of Cereal Science 43, 258-258.

Berland, S., and Launay, B. (1995). Rheological properties of wheat-flour doughs in steady and dynamic shear - effect of water-content and some additives. Cereal Chemistry 72, 48-52.

Bettge, A., Rubenthaler, G. L., and Pomeranz, Y. (1989). Alveograph algorithms to predict functional-properties of wheat in bread and cookie baking. Cereal Chemistry 66, 81-86.

Bloksma, A. H. (1962). Slow creep of wheat flour doughs. Rheologica Acta 2, 217-230.

Bloksma, A. H. (1990a). Dough Structure, Dough Rheology, and Baking Quality. Cereal Foods World 35, 237-244.

Bloksma, A. H. (1990b). Rheology of the breadmaking process. Cereal Foods World 35, 228-236.

Bloksma, A. H., and Bushuk, W. (1988). Rheology and chemistry of dough. In "Wheat: Chemistry and Technology" (Y. Pomeranz, ed.), Vol. 2, pp. 131-217. AACC, St. Paul.

Bohlin, L., and Carlson, T. L. G. (1980). Dynamic viscoelastic properties of wheat-flour dough - Dependence on mixing time. Cereal Chemistry 57, 174-177.

Bushuk, W. (1998). Interactions in wheat doughs. In "Interactions: the keys to cereal quality" (R. J. Hamer and R. C. Hoseney, eds.), pp. 1-15. AACC, St. Paul.

Campos, D. T., Steffe, J. F., and Ng, P. K. W. (1997). Rheological behavior of undeveloped and developed wheat dough. Cereal Chemistry 74, 489-494.

Cauvain, S. P. (2007). Breadmaking processes. In "Technology of breadmaking" (S. P. Cauvain and L. S. Young, eds.), pp. 21-49. Springer, New York.

Cauvain, S. P., and Young, L. S. (2007). Technology of breadmaking. pp. 397. Springer, New York.

Chin, I. L., and Campbell, G. M. (2005a). Dough aeration and rheology: Part 1. Effects of mixing speed and headspace pressure on mechanical development of bread dough. Journal of the Science of Food and Agriculture 85, 2184-2193.

Chin, N. L., and Campbell, G. M. (2005b). Dough aeration and rheology: Part 2. Effects of flour type, mixing speed and total work input on aeration and rheology of bread dough. Journal of the Science of Food and Agriculture 85, 2194-2202.

Chuang, G. C. C., and Yeh, A. I. (2006a). Creep deformation modeling of glutinous rice cakes (mochi). Cereal Chemistry 83, 179-187.

Chuang, G. C. C., and Yeh, A. I. (2006b). Rheological characteristics and texture attributes of glutinous rice cakes (mochi). Journal of Food Engineering 74, 314-323.

Clarke, C. I., Schober, T. J., Dockery, P., O'Sullivan, K., and Arendt, E. K. (2004). Wheat sourdough fermentation: effects of time and acidification on fundamental rheological properties. Cereal Chemistry 81, 409-417.

Courtin, C. M., and Delcour, J. A. (2002). Arabinoxylans and endoxylanases in wheat flour bread-making. Journal of Cereal Science 35, 225-243.

D'Appolonia, B. L., and Kunerth, W. H. (1984). "The farinograph handbook," 3th/Ed. AACC, St.Paul.

Davidou, S., Michon, C., Ben Thabet, I., and Launay, B. (2008). Influence of shaping and orientation of structures on rheological properties of wheat flour dough measured in dynamic shear and in biaxial extension. Cereal Chemistry 85, 403-408.

De Graef, V. (2009). Microstructural properties of isothermal palm oil crystallization, Ghent University, Ghent.

De Leyn, I. (2006). Functional additives. In "Bakery products: science and technology" (Y. H. Hui, ed.), pp. 233-242. Blackwell Publishing, Ames, USA.

Delcour, J. A., and Hoseney, R. C. (2010). "Principles of cereal science and technology," 3th/Ed. AACC Intenational, St. Paul.

Dewettinck, K., Van Bockstaele, F., Kuhne, B., de Walle, D. V., Courtens, T. M., and Gellynck, X. (2008). Nutritional value of bread: Influence of processing, food interaction and consumer perception. Journal of Cereal Science 48, 243-257.

Dexter, J. E., Preston, K. R., Martin, D. G., and Gander, E. J. (1994). The effects of protein content and starch damage on the physical dough properties and bread-making quality of Canadian durum wheat. Journal of Cereal Science 20, 139-151.

Dobraszczyk, B. J. (2005). Course notes. In "Science of baking, April 6-8th ", Reading, UK.

Dobraszczyk, B. J., and Morgenstern, M. (2003). Rheology and the breadmaking process. Journal of Cereal Science 38, 229-245.

Dobraszczyk, B. J., and Roberts, C. A. (1994). Strain-hardening and dough gas cell-wall failure in biaxial extension. Journal of Cereal Science 20, 265-274.

Dobraszczyk, B. J., and Salmanowicz, B. P. (2008). Comparison of predictions of baking volume using large deformation rheological properties. Journal of Cereal Science 47, 292-301.

Dobraszczyk, B. J., Salmanowicz, B. P., Lugowska, B., and Chelkowski, J. (2005). Rapid quality assessment of wheat cultivars registered in Poland using the 2-g mixograph and multivariate statistical analysis. Cereal Chemistry 82, 182-186.

Dobraszczyk, B. J., Smewing, J., Albertini, M., Maesmans, G., and Schofield, J. D. (2003). Extensional rheology and stability of gas cell walls in bread doughs at elevated temperatures in relation to breadmaking performance. Cereal Chemistry 80, 218-224.

Don, C., Lichtendonk, W. J., Plijter, J. J., and Hamer, R. J. (2003a). Glutenin macropolymer: a gel formed by glutenin particles. Journal of Cereal Science 37, 1-7.

Don, C., Lichtendonk, W. J., Plijter, J. J., and Hamer, R. J. (2003b). Understanding the link between GMP and dough: from glutenin particles in flour towards developed dough. Journal of Cereal Science 38, 157-165.

Don, C., Lichtendonk, W. J., Plijter, J. J., van Vliet, T., and Hamer, R. J. (2005). The effect of mixing on glutenin particle properties: aggregation factors that affect gluten function in dough. Journal of Cereal Science 41, 69-83.

Dowell, F. E., Maghirang, E. B., Pierce, R. O., Lookhart, G. L., Bean, S. R., Xie, F., Caley, M. S., Wilson, J. D., Seabourn, B. W., Ram, M. S., Park, S. H., and Chung, O. K. (2008). Relationship of bread quality to kernel, flour, and dough properties. Cereal Chemistry 85, 82-91.

Dreese, P. C., Faubion, J. M., and Hoseney, R. C. (1988). Dynamic rheological properties of flour, gluten, and gluten-starch doughs. 2. Effect of various processing and ingredient changes. Cereal Chemistry 65, 354-359.

Dubois, M., Dubat, A., and Launay, B. (2008). "The alveoconsistograph handbook," 2nd ed. AACC.

Dunnewind, B., Sliwinski, E. L., Grolle, K., and van Vliet, T. (2004). The Kieffer dough and gluten extensibility rig - an experimental evaluation. Journal of Texture Studies 34, 537-560.

Edwards, N. M., Dexter, J. E., and Scanlon, M. G. (2002). Starch participation in durum dough linear viscoelastic properties. Cereal Chemistry 79, 850-856.

Edwards, N. M., Dexter, J. E., Scanlon, M. G., and Cenkowski, S. (1999). Relationship of creep-recovery and dynamic oscillatory measurements to durum wheat physical dough properties. Cereal Chemistry 76, 638-645.

Edwards, N. M., Mulvaney, S. J., Scanlon, M. G., and Dexter, J. E. (2003). Role of gluten and its components in determining durum semolina dough viscoelastic properties. Cereal Chemistry 80, 755-763.

Edwards, N. M., Peressini, D., Dexter, J. E., and Mulvaney, S. J. (2001). Viscoelastic properties of durum wheat and common wheat dough of different strengths. Rheologica Acta 40, 142-153.

Eliasson, A. C., and Larsson, K. (1993). "Cereals in breadmaking: a molecular colloidal approach," Marcel Dekker, New York.

Engmann, J., Peck, M. C., and Wilson, D. I. (2005). An experimental and theoretical investigation of bread dough sheeting. Food and Bioproducts Processing 83, 175-184.

Esselink, E., van Aalst, H., Maliepaard, M., Henderson, T. M. H., Hoekstra, N. L. L., and van Duynhoven, J. (2003). Impact of industrial dough processing on structure: a rheology, nuclear magnetic resonance, and electron microscopy study. Cereal Chemistry 80, 419-423.

Faergestad, E. M., Molteberg, E. L., and Magnus, E. M. (2000). Interrelationships of protein composition, protein level, baking process and the characteristics of hearth bread and pan bread. Journal of Cereal Science 31, 309-320.

FAOSTAT (2011). www.faostat.fao.org (consulted januari 2011).

Faubion, J. M., Dreese, P. C., and Diehl, K. C. (1985). Dynamic rheological testing of wheat flour doughs. In "Rheology of wheat products" (H. Faridi, ed.), pp. 91-116. AACC, St. Paul.

Finney, K. F., and Barmore, M. A. (1948). Loaf volume and protein content of hard winter and spring wheats. Cereal Chemistry 25, 291-312.

Foubert, I. (2003). Modelling isothermal cocoa butter crystallization: influence of temperature and chemical composition. Ph.D thesis, Ghent University, Ghent.

Foubert, I., Vanrolleghem, P., Thas, O., and Dewettinck, K. (2004). Influence of chemical composition on the isothermal cocoa butter crystallization. Journal of Food Science 69, E478-E487.

Foubert, I., Vanrolleghem, P. A., and Dewettinck, K. (2003). A differential scanning calorimetry method to determine the isothermal crystallization kinetics of cocoa butter. Thermochimica Acta 400, 131-142.

Gan, Z., Ellis, P. R., and Schofield, J. D. (1995). Gas cell stabilisation and gas retention in wheat bread dough. Journal of Cereal Science 21, 215-230.

Gianibelli, C., Larroque, O. R., MacRitchie, F., and Wrigley, C. W. (2001). Biochemical, genetic and molecular characterization of wheat glutenin and its component subunits. Cereal Chemistry 78, 635-646.

Goesaert, H., Brijs, K., Veraverbeke, W. S., Courtin, C. M., Gebruers, K., and Delcour, J. A. (2005). Wheat flour constituents: how they impact bread quality, and how to impact their functionality. Trends in Food Science & Technology 16, 12-30.

Gooding, M. J. (2009). The wheat crop. In "Wheat Chemistry and Technology" (K. Khan and P. R. Shewry, eds.), pp. 19-49. AACC International, St. Paul.

Gras, P. W., Carpenter, H. C., and Anderssen, R. S. (2000). Modelling the developmental rheology of wheat-flour dough using extension tests. Journal of Cereal Science 31, 1-13.

Graveland, A, Bosveld, P., Lichtendonk, W. J., Marseille, J. P., Moonen, J. H. E. and Scheepstra, A. (1985). A model for the molecular-structure of the glutenins from wheat flour. Journal of Cereal Science 3, 1-16.

Greenwood, C. T. and Ewart, J. A. D. (1975). Hypothesis for structure of glutenin in relation to rheological properties of gluten and dough. Cereal Chemistry 52, 146-153.

Gunasekaran, S., and Ak, M. M. (2000). Dynamic oscillatory shear testing of foods -- selected applications. Trends in Food Science & Technology 11, 115-127.

Haegens, N. (2006). Mixing, dough making and dough makeup. In "Bakery products: science and technology" (Y. H. Hui, ed.), pp. 245-259. Blackwell Publishing, Ames.

Hamer, R. J., MacRitchie, F., and Weegels, P. L. (2009). Structure and functional properties of gluten. In "Wheat Chemistry and Technology" (K. Khan and P. R. Shewry, eds.), pp. 153-178. AACC International, St. Paul.

Hamer, R. J., and van Vliet, T. (2000). Understanding the structure and properties of gluten: an overview. In "7th International gluten workshop" (P. R. Shewry and A. S. Thatam, eds.), pp. 125-131. Royal Society of Chemistry.

Hamer, R. J., van Vliet, T., and Lefebvre, J. (2005). Letter to the Editor. Journal of Cereal Science 42, 344-345.

Haraszi, R., Larroque, O. R., Butow, B. J., Gale, K. R., and Bekes, F. (2008). Differential mixing action effects on functional properties and polymeric protein size distribution of wheat dough. Journal of Cereal Science 47, 41-51.

Hayman, D., Sipes, K., Hoseney, R. C., and Faubion, J. M. (1998). Factors controlling gas cell failure in bread dough. Cereal Chemistry 75, 585-589.

He, H., and Hoseney, R. C. (1991). Differences in gas retention, protein solubility, and rheological properties between flours of different baking quality. Cereal Chemistry 68, 526-530.

Hibberd, G. E. (1970a). Dynamic viscoelastic behaviour of wheat flour doughs. Part II: effect of water content in the linear region. Rheologica Acta 9, 497-500.

Hibberd, G. E. (1970b). Dynamic viscoelastic behaviour of wheat flour doughs. Part III: the influence of the starch granules. Rheologica Acta 9, 501-505.

Hibberd, G. E., and Parker, N. S. (1979). Non-linear creep and creep recovery of wheat flour doughs. Cereal Chemistry 56, 232-236.

Hibberd, G. E., and Wallace, W. J. (1966). Dynamic viscoelastic behaviour of wheat flour doughs. Part 1: linear aspects. Rheologica Acta 5, 193-198.

Hoseney, R. C. (1992). "Principles of cereal science and technology", pp327, AACC, Minnesota, USA.

Houben, A., Gotz, H., Mitzscherling, M., and Becker, T. (2010). Modification of the rheological behavior of amaranth (*Amaranthus hypochondriacus*) dough. Journal of Cereal Science 51, 350-356.

ICC. 2005. Standard methods of the International Association for Cereal Science and Technology (ICC). ICC: Vienna.

Janssen, A. M., vanVliet, T., and Vereijken, J. M. (1996a). Fundamental and empirical rheological behaviour of wheat flour doughs and comparison with bread making performance. Journal of Cereal Science 23, 43-54.

Janssen, A. M., vanVliet, T., and Vereijken, J. M. (1996b). Rheological behaviour of wheat glutens at small and large deformations. Comparison of two glutens differing in bread making potential. Journal of Cereal Science 23, 19-31.

Jongen, T. R. G., Bruschke, M. V., and Dekker, J. G. (2003). Analysis of dough kneaders using numerical flow simulations. Cereal Chemistry 80, 383-389.

Joye, I. J., Lagrain, B., and Delcour, J. A. (2009). Use of chemical redox agents and exogenous enzymes to modify the protein network during breadmaking - A review. Journal of Cereal Science 50, 11-21.

Kasarda, D. D., Woodard, K. M., and Adalsteins, A. E. (1998). Resolution of high molecular weight glutenin subunits by a new SDS-PAGE system incorporating a neutral pH buffer. Cereal Chemistry 75, 70-71.

Kawai, H., Tanaka, F., Takahashi, H., Hashimoto, N., and Yamauchi, H. (2006). Relationship between physical properties of dough and expansion ability during bread-making. Food Science and Technology Research 12, 91-95.

Kenny, S., Wehrle, K., Dennehy, T., and Arendt, E. K. (1999). Correlations between empirical and fundamental rheology measurements and baking performance of frozen bread dough. Cereal Chemistry 76, 421-425.

Khan, K., Tamminga, G. and Lukow, O. (1989). The effect of wheat flour proteins on mixing and baking – correlations with protein fractions and high molecular wheight glutenin subunit composition by gel electrophoresis. Cereal Chemistry 66, 391-396.

Khatkar, B. S. (2004). Effect of mixing time on dynamic rheological properties of wheat flour dough. Journal of Food Science and Technology-Mysore 41, 320-322.

Khatkar, B. S., Bell, A. E., and Schofield, J. D. (1995). The dynamic rheological properties of glutens and gluten sub-fractions from wheats of good and poor bread making quality. Journal of Cereal Science 22, 29-44.

Khatkar, B. S., and Schofield, J. D. (2002). Dynamic rheology of wheat flour dough. II. Assessment of dough strength and bread-making quality. Journal of the Science of Food and Agriculture 82, 823-826.

Kieffer, R., Garnreiter, F., and Belitz, H. D. (1981). Beurteilung von teigeigenschaften durch zugversuche im mikromaβstab. Zeitschrift fur lebensmittel-untersuchung und -forschung 172, 193-194.

Kieffer, R., Wieser, H., Henderson, M. H., and Graveland, A. (1998). Correlations of the breadmaking performance of wheat flour with rheological measurements on a micro-scale. Journal of Cereal Science 27, 53-60.

Kilborn, R. H., and Tipples, K. H. (1972). Factors affecting mechanical dough development. 1. Effect of mixing intensity and work input. Cereal Chemistry 49, 34-&.

Kim, Y. R., Cornillon, P., Campanella, O. H., Stroshine, R. L., Lee, S., and Shim, J. Y. (2008). Small and large deformation rheology for hard wheat flour dough as influenced by mixing and resting. Journal of Food Science 73, E1-E8.

Kokelaar, J. J., van Vliet, T., and Prins, A. (1996). Strain hardening properties and extensibility of flour and gluten doughs in relation to breadmaking performance. Journal of Cereal Science 24, 199-214.

Konopka, I., Fornal, L., Abramczyk, D., Rothkaehl, J., and Rotkiewicz, D. (2004). Statistical evaluation of different technological and rheological tests of Polish wheat varieties for bread volume prediction. International Journal of Food Science and Technology 39, 11-20.

Ktenioudaki, A., Butler, F., and Gallagher, E. (2010a). The effect of different mixing processes on dough extensional rheology and baked attributes. Vol. 90, pp. 2098-2104.

Ktenioudaki, A., Butler, F., and Gallagher, E. (2010b). Rheological properties and baking quality of wheat varieties from various geographical regions. Journal of Cereal Science 51, 402-408.

Lazaridou, A., Duta, D., Papageorgiou, M., Belc, N., and Biliaderis, C. G. (2007). Effects of hydrocolloids on dough rheology and bread quality parameters in gluten-free formulations. Journal of Food Engineering 79, 1033-1047.

Lefebvre, J. (2006). An outline of the non-linear viscoelastic behaviour of wheat flour dough in shear. Rheologica Acta 45, 525-538.

Lefebvre, J. (2009). Nonlinear, time-dependent shear flow behaviour, and shear-induced effects in wheat flour dough rheology. Journal of Cereal Science 49, 262-271.

Lefebvre, J., and Mahmoudi, N. (2007). The pattern of the linear viscoelastic behaviour of wheat flour dough as delineated from the effects of water content and high molecular weight glutenin subunits composition. Journal of Cereal Science 45, 49-58.

Letang, C., Piau, M., and Verdier, C. (1999). Characterization of wheat flour-water doughs. Part I: Rheometry and microstructure. Journal of Food Engineering 41, 121-132.

Lindahl, L., and Eliasson, A. C. (1992). A comparison of some rheological properties of durum and wheat-flour doughs. Cereal Chemistry 69, 30-34.

Lynch, E. J., Dal Bello, F., Sheehan, E. M., Cashman, K. D., and Arendt, E. K. (2009). Fundamental studies on the reduction of salt on dough and bread characteristics. Food Research International 42, 885-891.

MacRitchie, F. (2003). Fundamentals of dough formation. Cereal Foods World 48, 173-176.

MacRitchie, F., and Gras, P. W. (1973). The role of flour lipids in baking. Cereal Chemistry 50, 292-302.

Mani, K., Eliasson, A. C., Lindahl, L., and Tragardh, C. (1992). Rheological properties and breadmaking quality of wheat-flour doughs made with different dough mixers. Cereal Chemistry 69, 222-225.

Marsh, D., and Cauvain, S. P. (2007). Mixing and dough processing. In "Technology of breadmaking" (S. P. Cauvain and L. S. Young, eds.), pp. 93-140. Springer, New York.

Martin, P. J., Chin, N. L., Campbell, G. M., and Morrant, C. J. (2004). Aeration during bread dough mixing III. Effect of scale-up. Food and Bioproducts Processing 82, 282-290.

Masi, P., Cavella, S., and Sepe, M. (1998). Characterization of dynamic viscoelastic behavior of wheat flour doughs at different moisture contents. Cereal Chemistry 75, 428-432.

Mezger, T. G. (2002). "The rheology handbook: for users of rotational and oscillation rheometers," Vincentz Verlag, Hannover.

Miller, K. A., and Hoseney, R. C. (1999). Effect of oxidation on the dynamic rheological properties of wheat flour-water doughs. Cereal Chemistry 76, 100-104.

Miller, R. A., and Hoseney, R. C. (2008). Role of salt in baking. Cereal Foods World 53, 4-6.

Mitsoulis, E., and Hatzikiriakos, S. G. (2009). Rolling of bread dough: Experiments and simulations. Food and Bioproducts Processing 87, 124-138.

Moreira, R., Chenlo, F., Torres, M. D., and Prieto, D. M. (2010). Influence of the particle size on the rheological behaviour of chestnut flour doughs. Journal of Food Engineering 100, 270-277.

Nash, D., Lanning, S. P., Fox, P., Martin, J. M., Blake, N. K., Souza, E., Graybosch, R. A., Giroux, M. J., and Talbert, L. E. (2006). Relationship of dough extensibility to dough strength in a spring wheat cross. Cereal Chemistry 83, 255-258.

Navickis, L. L., Anderson, R. A., Bagley, E. B., and Jasberg, B. K. (1982). Viscoelastic properties of wheat flour doughs: variation of dynamic moduli with water and protein content. Journal of Texture Studies 13, 249-264.

Nunes, M. H. B., Ryan, L. A. M., and Arendt, E. K. (2009). Effect of low lactose dairy powder addition on the properties of gluten-free batters and bread quality. European Food Research and Technology 229, 31-41.

Onyango, C., Mutungi, C., Unbehend, G., and Lindhauer, M. G. (2010). Rheological and baking characteristics of batter and bread prepared from pregelatinised cassava starch and sorghum and modified using microbial transglutaminase. Journal of Food Engineering 97, 465-470.

Onyango, C., Unbehend, G., and Lindhauer, M. G. (2009). Effect of cellulose-derivatives and emulsifiers on creep-recovery and crumb properties of gluten-free bread prepared from sorghum and gelatinised cassava starch. Food Research International 42, 949-955.

Osborne, T. B. (1907). The protein of the wheat kernel. Publication No. 84. Carnegie Institute: Washington, DC.

Park, S. H., Bean, S. R., Chung, O. K., and Seib, P. A. (2006). Levels of protein and protein composition in hard winter wheat flours and the relationship to breadmaking. Cereal Chemistry 83, 418-423.

Payne, P. I., Corfield, K. G., Holt, L. M. and Blackman, J. A. (1981). Correlations between the inheritance of certain high-molecular weight subunits of glutenin and bread-making quality in progenies of six crosses of bread wheat. Journal of the Science of Food and Agriculture 32, 51-60.

Payne, P. I. and Lawrence, G. J. (1983). Catalogue of alleles for the complex gene loci, Glu-A1, Glu-B1, and Glu-D1 which code for high-molecular weight subunits of glutenin in hexaploid wheat. Cereal Research Communications 11, 29-35.

Payne, P. I., Nightingale, M. A., Krattiger, A. F. and Holt, L. M. 1987. The relationship between HMW glutenin subunit composition and the bread-making quality of British-grown wheat varieties. Journal of the Science of Food and Agriculture 40, 51-65.

Pedersen, L., and Jorgensen, J. R. (2007). Variation in rheological properties of gluten from three biscuit wheat cultivars in relation to nitrogen fertilisation. Journal of Cereal Science 46, 132-138.

Pedersen, L., Kaack, K., Bergsoe, M. N., and Adler-Nissen, J. (2004). Rheological properties of biscuit dough from different cultivars, and relationship to baking characteristics. Journal of Cereal Science 39, 37-46.

Peighambardoust, S. H., van der Goot, A. J., Hamer, R. J., and Boom, R. M. (2004). A new method to study simple shear processing of wheat gluten-starch mixtures. Cereal Chemistry 81, 714-721.

Peighambardoust, S. H., van der Goot, A. J., van Vliet, T., Hamer, R. J., and Boom, R. M. (2006). Microstructure formation and rheological behaviour of dough under simple shear flow. Journal of Cereal Science 43, 183-197.

Peña, E., Bernardo, A., Soler, C., and Jouve, N. (2006). Do tyrosine crosslinks contribute to the formation of the gluten network in common wheat (*Triticum aestivum L.*) dough? Journal of Cereal Science 44, 144-153.

Peressini, D., Peighambardoust, S. H., Hamer, R. J., Sensidoni, A., and van der Goot, A. J. (2008). Effect of shear rate on microstructure and rheological properties of sheared wheat doughs. Journal of Cereal Science 48, 426-438.

Qi, F. Z., Dai, S. C., Newberry, M. P., Love, R. J., and Tanner, R. I. (2008). A simple approach to predicting dough sheeting thickness. Journal of Cereal Science 47, 489-495.

Rasper, V. F. (1976). Texture of dough, pasta and baked products. In: "Rheology & Texture in Food Quality". (De Man J. M., Voisly, P. W., Rasper, V. F. and Stanley, D. W., eds.), pp 308-354. AVI: Westport.

Roels, S. P., Cleemput, G., Vandewalle, X., Nys, M., and Delcour, J. A. (1993). Bread volume potential of variable-quality flours with constant protein level as determined by factors governing mixing time and baking absorption levels. Cereal Chemistry 70, 318-323.

Ross, K. A., Pyrak-Nolte, L. J., and Campanella, O. H. (2004). The use of ultrasound and shear oscillatory tests to characterize the effect of mixing time on the rheological properties of dough. Food Research International 37, 567-577.

Rouille, J., Della Valle, G., Lefebvre, J., Sliwinski, E., and van Vliet, T. (2005). Shear and extensional properties of bread doughs affected by their minor components. Journal of Cereal Science 42, 45-57.

Safari-Ardi, M., and Phan-Thien, N. (1998). Stress relaxation and oscillatory tests to distinguish between doughs prepared from wheat flours of different varietal origin. Cereal Chemistry 75, 80-84.

Salvador, A., Sanz, T., and Fiszman, S. M. (2006). Dynamic rheological characteristics of wheat flour-water doughs. Effect of adding NaCl, sucrose and yeast. Food Hydrocolloids 20, 780-786.

Sasaki, T., Yasui, T., and Kohyama, K. (2008). Influence of starch and gluten characteristics on rheological properties of wheat flour gel at small and large deformation. Cereal Chemistry 85, 329-334.

Schluentz, E. J., Steffe, J. F., and Ng, P. K. W. (2000). Rheology and microstructure of wheat dough developed with controlled deformation. Journal of Texture Studies 31, 41-54.

Schober, T. J., Clarke, C. I., and Kuhns, M. (2002). Characterization of functional properties of gluten proteins in spelt cultivars using rheological and quality factor measurements. Cereal Chemistry 79, 408-417.

Schober, T. J., Dockery, P., and Arendt, E. K. (2003). Model studies for wheat sourdough systems using gluten, lactate buffer and sodium chloride. European Food Research and Technology 217, 235-243.

Shewry, P. R., D'Ovidio, R., Lafiandra, D., Jenkins, J., Mills, E. N. C., and Békés, F. (2009). Wheat grain proteins. In "Wheat Chemistry and Technology" (K. Khan and P. R. Shewry, eds.), pp 223-298. AACC International, St.Paul.

Shewry, P. R., Popineau, Y., Lafiandra, D., and Belton, P. (2000). Wheat glutenin subunits and dough elasticity: findings of the EUROWHEAT project. Trends in Food Science & Technology 11, 433-441.

Singh, H., and MacRitchie, F. (2001). Application of polymer science to properties of gluten. Journal of Cereal Science 33, 231-243.

Sivaramakrishnan, H. P., Senge, B., and Chattopadhyay, P. K. (2004). Rheological properties of rice dough for making rice bread. Journal of Food Engineering 62, 37-45.

Skendi, A., Papageorgiou, M., and Biliaderis, C. G. (2010). Influence of water and barley beta-glucan addition on wheat dough viscoelasticity. Food Research International 43, 57-65.

Sliwinski, E. L., Kolster, P., and van Vliet, T. (2004a). Large-deformation properties of wheat dough in uni- and biaxial extension. Part I. Flour dough. Rheologica Acta 43, 306-320.

Sliwinski, E. L., Kolster, P., and van Vliet, T. (2004b). On the relationship between large-deformation properties of wheat flour dough and baking quality. Journal of Cereal Science 39, 231-245.

Sliwinski, E. L., van der Hoef, F., Kolster, P., and van Vliet, T. (2004c). Large-deformation properties of wheat dough in uni- and biaxial extension. Part II. Gluten dough. Rheologica Acta 43, 321-332.

Smewing, J. (1995). "The measurement of dough and gluten extensibility using the SMS/Kieffer rig and TA.XT2 texture analyzer" Stable Micro Systems Ltd., Surray, UK.

Smith, J. R., Smith, T. L., and Tschoegl, N. W. (1970). Rheological properties of wheat flour doughs. III. Dynamic shear modulus and its dependence on amplitude, frequency and dough composition. Rheologica Acta 9, 239-252.

Smith, T. L., and Tschoegl, N. W. (1970). Rheological properties of wheat flour doughs. IV. Creep and creep recovery in simple tension. Rheologica Acta 9, 339-344.

Song, Y. H., and Zheng, Q. (2007). Dynamic rheological properties of wheat flour dough and proteins. Trends in Food Science & Technology 18, 132-138.

Sozer, N. (2009). Rheological properties of rice pasta dough supplemented with proteins and gums. Food Hydrocolloids 23, 849-855.

Sroan, B. S., Bean, S. R., and MacRitchie, F. (2009). Mechanism of gas cell stabilization in bread making. I. The primary gluten-starch matrix. Journal of Cereal Science 49, 32-40.

Sroan, B. S., and MacRitchie, F. (2009). Mechanism of gas cell stabilization in breadmaking. II. The secondary liquid lamellae. Journal of Cereal Science 49, 41-46.

Stauffer, C. E. (2007). Principles of dough formation. In "Technology of breadmaking" (S. P. Cauvain and L. S. Young, eds.), pp. 299-332. Springer, New York.

Steffe, J. F. (1996). "Rheological methods in food engineering," 2nd/Ed. Freeman Press, East Lansing.

Stojceska, V., Butler, F., Gallagher, E., and Keehan, D. (2007). A comparison of the ability of several small and large deformation rheological measurements of wheat dough to predict baking behaviour. Journal of Food Engineering 83, 475-482.

Stone, B., and Morell, M. K. (2009). Carbohydrates. In "Wheat Chemistry and Technology" (K. Khan and P. R. Shewry, eds.), pp 299-362. AACC International, St.Paul.

Suchy, J., Lukow, O. M., and Ingelin, M. E. (2000). Dough microextensibility method using a 2-g mixograph and a texture analyzer. Cereal Chemistry 77, 39-43.

Tilley, K. A., Benjamin, R. E., Bagorogoza, K. E., Okot-Kotber, B. M., Prakash, O., and Kwen, H. (2001). Tyrosine cross-links: Molecular basis of gluten structure and function. Journal of Agricultural and Food Chemistry 49, 2627-2632.

Tipples, K. H., Meredith, J. O., and Holas, J. (1978). Factors affecting farinograph and baking absorption. 2. Relative influence of flour components. Cereal Chemistry 55, 652-660.

Tronsmo, K. M., Faergestad, E. M., Schofield, J. D., and Magnus, E. M. (2003a). Wheat protein quality in relation to baking performance evaluated by the Chorleywood bread process and a hearth bread baking test. Journal of Cereal Science 38, 205-215.

Tronsmo, K. M., Magnus, E. M., Baardseth, P., Schofield, J. D., Aamodt, A., and Faergestad, E. M. (2003b). Comparison of small and large deformation rheological properties of wheat dough and gluten. Cereal Chemistry 80, 587-595.

Tronsmo, K. M., Magnus, E. M., Faergestad, E. M., and Schofield, J. D. (2003c). Relationships between gluten rheological properties and hearth loaf characteristics. Cereal Chemistry 80, 575-586.

Tung, M. A., and Paulson, A. T. (1995). Rheological concepts for probing ingredient interactions in food systems. In "Ingredient Interactions: effects on food quality" (A. G. Gaonkar, ed.), pp. 45-83. Marcel Dekker Inc., New York.

Uthayakumaran, S., Newberry, M., Keentok, M., Stoddard, F. L., and Bekes, F. (2000). Basic rheology of bread dough with modified protein content and glutenin-to-gliadin ratios. Cereal Chemistry 77, 744-749.

Uthayakumaran, S., Newberry, M., Phan-Thien, N., and Tanner, R. (2002). Small and large strain rheology of wheat gluten. Rheologica Acta 41, 162-172.

van de Velde, F., Weinbreck, F., Edelman, M. W., van der Linden, E., and Tromp, R. H. (2003). Visualisation of biopolymer mixtures using confocal scanning laser microscopy (CSLM) and covalent labelling techniques. Colloids and Surfaces B: Biointerfaces 31, 159-168.

van der Zalm, E. E. J., van der Goot, A. J., and Boom, R. M. (2010). Influence of sodium chloride on shear flow induced starch-gluten separation from Soissons wheat dough. Journal of Food Engineering 99, 366-372.

van Vliet, T. (2008). Strain hardening as an indicator of bread-making performance: A review with discussion. Journal of Cereal Science 48, 1-9.

van Vliet, T., and Hamer, R. J. (2007). Letter to the editor. Journal of Cereal Science 46, 98-99.

Van Vliet, T., Janssen, A. M., Bloksma, A. H., and Walstra, P. (1992). Strain hardening of dough as a requirement for gas retention. Journal of Texture Studies 23, 439-460.

Vanneste, J., and De Leyn, I. (2004). Experiences with the simultaneous use in the baking test of a baking plate with U-profile and a normal baking pan. Getreidetechnologie 58, 286-291.

Veraverbeke, W. S., and Delcour, J. A. (2002). Wheat protein composition and properties of wheat glutenin in relation to breadmaking functionality. Critical Reviews in Food Science and Nutrition 42, 179-208.

Verbeken, D. (2006). Functionality of k-carrageenan in complex food gels. PhD, Ghent University, Ghent.

Wang, F. C., and Sun, X. S. (2002). Creep-recovery of wheat flour doughs and relationship to other physical dough tests and breadmaking performance. Cereal Chemistry 79, 567-571.

Weegels, P. L., Hamer, R. J., and Schofield, J. D. (1997). Depolymerisation and re-polymerisation of wheat glutenin during dough processing. 2. Changes in composition. Journal of Cereal Science 25, 155-163.

Wehrle, K., Grau, H., and Arendt, E. K. (1997). Effects of lactic acid, acetic acid, and table salt on fundamental rheological properties of wheat dough. Cereal Chemistry 74, 739-744.

Weipert, D. (1990). The Benefits of Basic Rheometry in Studying Dough Rheology. Cereal Chemistry 67, 311-317.

Whorlow, R. (1992). "Rheological techniques," 2nd/Ed. Ellis Horwood Ltd., New York.

Wikstrom, K., and Bohlin, L. (1999). Extensional flow studies of wheat flour dough. I. Experimental method for measurements in contraction flow geometry and application to flours varying in breadmaking performance. Journal of Cereal Science 29, 217-226.

Wrigley, C. W., Bekes, F., and Bushuk, W. (2006). Gluten: a balance of gliadin and glutenin. In "Gliadin and glutenin: the unique balance of wheat quality" (C. W. Wrigley, F. Bekes and W. Bushuk, eds.), pp. 3-32. AACC International, St. Paul.

Wu, M., Li, D., Wang, L. J., Ozkan, N., and Mao, Z. H. (2010). Rheological properties of extruded dispersions of flaxseed-maize blend. Journal of Food Engineering 98, 480-491.

Zheng, H., Morgenstern, M. P., Campanella, O. H., and Larsen, N. G. (2000). Rheological properties of dough during mechanical dough development. Journal of Cereal Science 32, 293-306.

Annexes

BROIZEL: kruimel, in: -liever een brok as een broizel-:
liever een groot stuk dan een klein.

[Oilsjtersen Diksjoneir]

Annex 1. Empirical rheological evaluation (Farinograph and Alveograph) of the 17 wheat flours [a]

	Farinograph						Alveograph				
	WA	**DDT**	**STAB**	**SOFT**	**QUAL**	**ELAST**	**P**	**L**	**W**	**P/L**	**I_e**
Cultivar	(%)	(min)	(min)	(FU)	(-)	(FU)	(mmH_2O)	(mm)	(10^{-4} J)	(-)	(%)
Akteur	58.8 ± 0.4	6.6 ± 0.1	11.9 ± 0.7	55.3 ± 3.2	122.3 ± 7.1	95.3 ± 4.9	63.3 ± 0.6	159.3 ± 4.6	285.7 ± 8.1	0.40 ± 0.01	57.3 ± 0.2
Altos	63.3 ± 0.3	3.6 ± 0.1	10.2 ± 0.7	51.7 ± 4.2	107.3 ± 12.1	82.3 ± 3.8	125.8 ± 3.4	74.0 ± 0.0	318.4 ± 8.4	1.70 ± 0.05	53.2 ± 0.4
Anthus	58.8 ± 0.0	4.6 ± 0.1	5.8 ± 0.2	77.3 ± 0.6	74.7 ± 2.1	79.3 ± 3.8	71.8 ± 2.4	97.7 ± 8.7	186.2 ± 14.1	0.74 ± 0.09	43.3 ± 3.6
Astuce	50.1 ± 0.4	1.9 ± 0.2	12.6 ± 1.2	39.7 ± 4.5	35.3 ± 0.6	89.3 ± 9.7	42.5 ± 2.2	118.3 ± 5.8	162.3 ± 12.6	0.37 ± 0.03	55.0 ± 1.3
Bussard	63.7 ± 0.1	7.4 ± 0.1	11.4 ± 0.3	55.0 ± 2.6	127.3 ± 5.5	114.7 ± 3.5	67.7 ± 3.8	187.2 ± 7.5	311.4 ± 13.0	0.36 ± 0.03	52.9 ± 2.2
Constance	51.1 ± 0.1	6.3 ± 0.1	12.3 ± 1.8	43.3 ± 7.5	134.7 ± 22.2	91.3 ± 4.9	47.7 ± 1.2	124.3 ± 9.3	163.7 ± 8.1	0.39 ± 0.03	49.9 ± 0.5
Dinosor	54.5 ± 0.1	2.5 ± 0.2	18.1 ± 0.9	20.7 ± 5.8	179.3 ± 18.0	89.7 ± 5.1	74.0 ± 4.0	115.7 ± 8.6	243.7 ± 6.7	0.64 ± 0.08	51.2 ± 1.9
Enorm	58.9 ± 0.2	5.3 ± 0.1	16.4 ± 1.7	40.0 ± 4.4	147.7 ± 12.7	96.0 ± 5.6	94.3 ± 1.5	122.3 ± 5.0	352.7 ± 6.7	0.77 ± 0.03	57.3 ± 1.2
Ephoros	58.7 ± 0.2	4.7 ± 0.2	6.4 ± 0.7	79.0 ± 11.3	79.7 ± 11.3	81.3 ± 6.1	70.7 ± 0.6	101.3 ± 3.2	188.5 ± 8.8	0.70 ± 0.02	43.5 ± 1.7
Kodex	60.8 ± 0.3	5.4 ± 0.2	11.5 ± 1.6	49.7 ± 10.0	127.0 ± 17.8	92.7 ± 3.1	99.7 ± 2.4	78.3 ± 2.5	251.7 ± 11.8	1.27 ± 0.04	49.1 ± 2.2
Melkior	59.3 ± 0.0	8.8 ± 0.2	14.4 ± 0.6	45.3 ± 3.2	151.7 ± 9.7	89.0 ± 3.5	99.3 ± 3.5	94.3 ± 5.5	303.3 ± 14.0	1.06 ± 0.09	55.5 ± 1.0
Quebon	62.0 ± 0.1	6.0 ± 0.2	9.0 ± 0.9	59.7 ± 6.7	106.3 ± 4.2	81.3 ± 4.2	99.0 ± 1.0	97.7 ± 3.1	295.3 ± 3.8	1.01 ± 0.04	52.8 ± 0.3
Rosario	58.5 ± 0.3	4.9 ± 0.1	7.0 ± 0.4	72.3 ± 5.5	85.0 ± 5.3	84.7 ± 6.0	59.2 ± 6.5	141.7 ± 13.6	203.8 ± 10.7	0.42 ± 0.08	47.0 ± 1.7
Rustic	60.1 ± 0.3	4.0 ± 0.1	12.0 ± 0.6	46.0 ± 1.7	121.7 ± 1.2	69.3 ± 7.5	114.5 ± 5.2	78.1 ± 3.1	311.8 ± 12.6	1.50 ± 0.10	54.9 ± 3.2
Samurai	54.7 ± 0.2	3.9 ± 0.2	5.6 ± 0.6	77.7 ± 5.0	63.3 ± 5.0	93.7 ± 5.9	47.6 ± 2.7	160.3 ± 7.6	164.0 ± 14.0	0.30 ± 0.02	42.4 ± 2.6
Sokrates	58.2 ± 0.3	6.8 ± 0.2	11.4 ± 0.7	55.3 ± 3.8	122.0 ± 6.9	89.3 ± 4.2	80.8 ± 2.1	99.7 ± 3.0	267.0 ± 14.6	0.81 ± 0.03	57.5 ± 1.8
Tuareg	54.3 ± 0.2	3.2 ± 0.3	13.3 ± 0.8	38.0 ± 3.0	129.0 ± 8.5	95.7 ± 0.6	59.0 ± 2.6	129.0 ± 1.0	216.7 ± 12.7	0.46 ± 0.03	52.6 ± 1.0
Min.	*50.1*	*1.9*	*5.6*	*20.7*	*35.3*	*69.3*	*42.5*	*74.0*	*162.3*	*0.30*	*42.4*
Max.	*63.7*	*8.8*	*18.1*	*79.0*	*179.3*	*114.7*	*125.8*	*187.2*	*352.7*	*1.70*	*57.5*

[a] Mean and standard deviation of three repetitions are reported

Annex 2. Rheological parameters obtained from rotational rheometry for the 17 wheat flours[a]

	Dynamic oscillation				Creep-recovery 100Pa			Creep-recovery 250Pa		
	G'	G''	$\|G^*\|$	δ	$J_{c,max}$	$J_{r,max}$	recovery	$J_{c,max}$	$J_{r,max}$	recovery
Cultivar	(Pa)	(Pa)	(Pa)	(°)	(10^{-3} Pa^{-1})	(10^{-3} Pa^{-1})	(%)	(10^{-3} Pa^{-1})	(10^{-3} Pa^{-1})	(%)
Akteur	6734 ± 126	2938 ± 50	7347 ± 135	23.6 ± 0.1	3.16 ± 0.11	1.70 ± 0.06	53.4	6.24 ± 0.24	2.02 ± 0.02	32.1
Altos	11271 ± 233	4472 ± 92	12125 ± 250	21.6 ± 0.1	1.19 ± 0.06	0.71 ± 0.03	59.6	2.16 ± 0.10	1.08 ± 0.02	49.5
Anthus	9555 ± 192	3792 ± 76	10280 ± 206	21.6 ± 0.1	1.78 ± 0.11	1.01 ± 0.06	56.4	4.15 ± 0.17	1.52 ± 0.02	36.3
Astuce	24502 ± 198	7974 ± 74	25765 ± 201	18.0 ± 0.1	0.35 ± 0.02	0.21 ± 0.01	58.9	0.53 ± 0.03	0.28 ± 0.01	52.9
Bussard	7130 ± 182	3258 ± 72	7839 ± 195	24.6 ± 0.1	4.07 ± 0.22	1.87 ± 0.09	45.6	8.12 ± 0.13	2.02 ± 0.05	24.7
Constance	19066 ± 373	6675 ± 131	20201 ± 395	19.3 ± 0.1	0.52 ± 0.03	0.29 ± 0.01	55.7	0.90 ± 0.04	0.44 ± 0.02	49.1
Dinosor	14187 ± 134	5338 ± 37	15157 ± 137	20.6 ± 0.1	0.89 ± 0.01	0.56 ± 0.01	62.3	1.47 ± 0.11	0.85 ± 0.06	57.6
Enorm	10676 ± 269	4248 ± 104	11500 ± 287	21.7 ± 0.1	1.22 ± 0.05	0.73 ± 0.03	59.6	2.20 ± 0.08	1.13 ± 0.02	50.9
Ephoros	11739 ± 176	4759 ± 72	12739 ± 189	21.9 ± 0.1	1.39 ± 0.02	0.79 ± 0.01	56.7	2.64 ± 0.06	1.18 ± 0.01	44.5
Kodex	11530 ± 185	4528 ± 92	12387 ± 206	21.4 ± 0.1	1.21 ± 0.05	0.70 ± 0.02	57.7	2.10 ± 0.12	1.05 ± 0.03	49.9
Melkior	17002 ± 219	6035 ± 75	18040 ± 231	19.5 ± 0.1	0.58 ± 0.03	0.37 ± 0.02	63.8	0.86 ± 0.00	0.52 ± 0.01	60.4
Quebon	10370 ± 169	4186 ± 67	11245 ± 182	21.9 ± 0.1	1.29 ± 0.06	0.76 ± 0.03	58.8	2.31 ± 0.10	1.16 ± 0.04	49.8
Rosario	8654 ± 144	3504 ± 56	9226 ± 154	22.3 ± 0.1	2.07 ± 0.05	1.11 ± 0.02	53.3	4.54 ± 0.16	1.56 ± 0.03	34.1
Rustic	13575 ± 303	5022 ± 113	14473 ± 323	20.3 ± 0.1	0.81 ± 0.02	0.49 ± 0.01	60.2	1.24 ± 0.02	0.68 ± 0.00	54.4
Samurai	13564 ± 246	5073 ± 84	14481 ± 259	20.5 ± 0.1	0.88 ± 0.05	0.48 ± 0.04	54.9	2.00 ± 0.06	0.88 ± 0.02	43.7
Sokrates	11723 ± 504	4389 ± 177	12517 ± 534	20.5 ± 0.1	0.86 ± 0.02	0.54 ± 0.01	62.5	1.88 ± 0.06	1.02 ± 0.01	53.8
Tuareg	12890 ± 368	4614 ± 109	13693 ± 382	19.7 ± 0.1	1.03 ± 0.05	0.63 ± 0.02	61.2	1.42 ± 0.06	0.85 ± 0.03	59.1
Min.	*6734*	*2938*	*7347*	*18.0*	*0.35*	*0.21*	*45.6*	*0.53*	*0.28*	*24.7*
Max.	*24502*	*7974*	*25765*	*24.6*	*4.07*	*1.87*	*63.8*	*8.12*	*2.02*	*60.4*

[a] Mean and standard deviation of 3 (creep-recovery) or 6 (dynamic oscillation) repetitions are reported.

Annex 3. Anova statistics for the model to predict bread volume based on three parameters (protein content, WA and P/L) obtained by forward linear regression

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	,750[a]	,563	,534	42,045
2	,837[b]	,700	,657	36,067
3	,894[c]	,798	,752	30,675

a. Predictors: (Constant), PROTEIN

b. Predictors: (Constant), PROTEIN, WA

c. Predictors: (Constant), PROTEIN, WA, P_L

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	34163,476	1	34163,476	19,326	,001[a]
	Residual	26516,642	15	1767,776		
	Total	60680,118	16			
2	Regression	42468,861	2	21234,431	16,324	,000[b]
	Residual	18211,256	14	1300,804		
	Total	60680,118	16			
3	Regression	48447,326	3	16149,109	17,162	,000[c]
	Residual	12232,792	13	940,984		
	Total	60680,118	16			

a. Predictors: (Constant), PROTEIN

b. Predictors: (Constant), PROTEIN, WA

c. Predictors: (Constant), PROTEIN, WA, P_L

d. Dependent Variable: BAKINGVOLUME

Coefficients[a]

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	73,289	103,803		,706	,491
	PROTEIN	33,283	7,571	,750	4,396	,001
2	(Constant)	-184,855	135,521		-1,364	,194
	PROTEIN	20,871	8,143	,471	2,563	,023
	WA	7,372	2,918	,464	2,527	,024
3	(Constant)	-355,645	133,704		-2,660	,020
	PROTEIN	13,302	7,549	,300	1,762	,102
	WA	12,900	3,312	,812	3,895	,002
	P_L	-61,278	24,311	-,421	-2,521	,026

a. Dependent Variable: BAKINGVOLUME

Annex 4. Creep-recovery model parameters of 17 wheat cultivars obtained from the creep curve at a shear stress of 100 or 250 Pa[*,]**

Wheat cultivar	J_0 (10^{-4} Pa^{-1})		J_1 (10^{-4} Pa^{-1})		J_2 (10^{-3} Pa^{-1})		r_1 (s)		r_2 (s)		μ_0 (10^5 Pa.s)	
	100 Pa	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*
1 Acteur	1.28 ± 0.03	1.54 ± 0.03	3.74 ± 0.12	5.30 ± 0.17	0.74 ± 0.02	1.61 ± 0.11	0.89 ± 0.01	1.21 ± 0.02	24.4 ± 0.1	39.9 ± 0.9	1.50 ± 0.06	0.74 ± 0.02
2 Altos	0.71 ± 0.01	0.86 ± 0.02	1.65 ± 0.05	2.25 ± 0.08	0.30 ± 0.01	0.45 ± 0.02	0.71 ± 0.02	0.88 ± 0.02	21.2 ± 0.3	26.9 ± 0.8	4.41 ± 0.33	2.08 ± 0.10
3 Anthus	0.93 ± 0.04	1.18 ± 0.02	2.34 ± 0.11	3.58 ± 0.08	0.45 ± 0.02	1.00 ± 0.03	0.81 ± 0.02	1.14 ± 0.01	24.0 ± 0.8	37.6 ± 0.2	2.89 ± 0.22	1.09 ± 0.05
4 Astuce	0.32 ± 0.01	0.35 ± 0.02	0.51 ± 0.03	0.69 ± 0.03	0.09 ± 0.01	0.14 ± 0.01	0.58 ± 0.11	0.72 ± 0.02	21.1 ± 1.8	23.4 ± 0.3	15.54 ± 1.07	9.95 ± 0.57
5 Bussard	1.27 ± 0.05	1.49 ± 0.04	4.12 ± 0.17	5.63 ± 0.15	0.92 ± 0.07	1.98 ± 0.06	1.13 ± 0.08	1.43 ± 0.02	29.0 ± 1.8	42.6 ± 0.3	1.12 ± 0.06	0.54 ± 0.01
6 Constance	0.40 ± 0.01	0.46 ± 0.02	0.68 ± 0.04	1.05 ± 0.04	0.13 ± 0.01	0.22 ± 0.01	0.58 ± 0.11	0.89 ± 0.01	21.0 ± 1.5	26.4 ± 0.3	10.01 ± 0.54	5.51 ± 0.27
7 Dinosor	0.62 ± 0.01	0.73 ± 0.03	1.40 ± 0.01	1.90 ± 0.10	0.24 ± 0.01	0.37 ± 0.03	0.62 ± 0.01	0.76 ± 0.02	19.7 ± 0.2	25.6 ± 0.5	6.23 ± 0.06	3.45 ± 0.30
8 Enorm	0.74 ± 0.01	0.92 ± 0.01	1.69 ± 0.05	2.45 ± 0.04	0.31 ± 0.01	0.50 ± 0.01	0.72 ± 0.04	0.86 ± 0.02	21.4 ± 0.7	27.1 ± 0.4	4.29 ± 0.22	2.12 ± 0.09
9 Ephoros	0.74 ± 0.03	0.92 ± 0.01	1.91 ± 0.03	2.71 ± 0.04	0.35 ± 0.01	0.64 ± 0.02	0.81 ± 0.03	0.99 ± 0.01	23.1 ± 0.1	31.7 ± 0.4	3.70 ± 0.07	1.77 ± 0.05
10 Kodex	0.71 ± 0.02	0.87 ± 0.02	1.64 ± 0.05	2.29 ± 0.07	0.30 ± 0.01	0.48 ± 0.02	0.71 ± 0.04	0.87 ± 0.01	21.8 ± 0.7	28.8 ± 0.2	4.24 ± 0.18	2.21 ± 0.16
11 Melkior	0.46 ± 0.03	0.55 ± 0.01	0.94 ± 0.04	1.25 ± 0.01	0.15 ± 0.01	0.21 ± 0.00	0.57 ± 0.10	0.63 ± 0.00	18.7 ± 1.7	20.5 ± 0.1	9.79 ± 0.62	6.01 ± 0.02
12 Quebon	0.75 ± 0.03	0.89 ± 0.02	1.8 0± 0.07	2.43 ± 0.09	0.33 ± 0.01	0.49 ± 0.03	0.73 ± 0.04	0.90 ± 0.02	21.8 ± 0.4	28.6 ± 1.0	4.03 ± 0.20	1.95 ± 0.08
13 Rosario	0.98 ± 0.03	1.24 ± 0.03	2.56 ± 0.02	3.86 ± 0.12	0.50 ± 0.01	1.10 ± 0.05	0.84 ± 0.03	1.11 ± 0.01	24.4 ± 0.7	35.4 ± 0.3	2.38 ± 0.09	0.99 ± 0.03
14 Rustic	0.60 ± 0.01	0.68 ± 0.00	1.19 ± 0.04	1.58 ± 0.01	0.19 ± 0.01	0.27 ± 0.01	0.58 ± 0.06	0.67 ± 0.01	19.5 ± 1.1	22.8 ± 0.3	6.57 ± 0.26	3.88 ± 0.08
15 Samurai	0.57 ± 0.06	0.69 ± 0.02	1.16 ± 0.14	1.86 ± 0.05	0.23 ± 0.02	0.47 ± 0.01	0.74 ± 0.09	1.16 ± 0.02	23.6 ± 1.4	32.5 ± 0.4	5.96 ± 0.16	2.27 ± 0.08
16 Sokrates	0.63 ± 0.01	0.91 ± 0.01	1.35 ± 0.02	2.27 ± 0.05	0.22 ± 0.01	0.41 ± 0.01	0.54 ± 0.01	0.70 ± 0.02	18.4 ± 0.1	24.7 ± 0.4	6.40 ± 0.23	2.50 ± 0.10
17 Tuareg	0.74 ± 0.01	0.78 ± 0.02	1.54 ± 0.06	1.84 ± 0.07	0.26 ± 0.01	0.35 ± 0.02	0.62 ± 0.01	0.74 ± 0.02	20.0 ± 0.3	24.8 ± 0.6	5.22 ± 0.26	3.51 ± 0.15
Min.	*0.32*	*0.35*	*0.51*	*0.69*	*0.09*	*0.14*	*0.54*	*0.63*	*18.4*	*20.5*	*1.12*	*0.54*
Max.	*1.28*	*1.54*	*4.12*	*5.63*	*0.92*	*1.98*	*1.13*	*1.43*	*29.0*	*42.6*	*15.54*	*5.51*

[*] J_0: instantaneous compliance, J_1, J_2: retarded elastic compliances, r_1, r_2: retardation times, μ_0: steady state viscosity

[**] Mean and standard deviation based on three repetitions

Annex 5. Creep-recovery model parameters of 17 wheat cultivars obtained from the recovery curve (10 min) recorded after a creep phase (5min) at 100 or 250 Pa[*,]**

	Wheat cultivar	J_0 (10^{-4} Pa^{-1})		J_1 (10^{-4} Pa^{-1})		J_2 (10^{-3} Pa^{-1})		r_1 (s)		r_2 (s)	
		100 Pa	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*	*100 Pa*	*250 Pa*
1	Acteur	1.74 ± 0.05	1.79 ± 0.02	5.81 ± 0.19	6.86 ± 0.06	0.85 ± 0.03	1.06 ± 0.01	1.96 ± 0.01	2.08 ± 0.01	67.0 ± 0.3	58.6 ± 0.5
2	Altos	0.91 ± 0.02	1.12 ± 0.02	2.38 ± 0.08	3.54 ± 0.07	0.34 ± 0.01	0.56 ± 0.01	1.76 ± 0.04	2.04 ± 0.01	73.1 ± 1.2	65.2 ± 0.2
3	Anthus	1.19 ± 0.07	1.43 ± 0.02	3.41 ± 0.20	5.05 ± 0.07	0.49 ± 0.03	0.79 ± 0.01	1.77 ± 0.02	2.11 ± 0.01	67.7 ± 0.8	61.1 ± 0.3
4	Astuce	0.38 ± 0.01	0.44 ± 0.02	0.64 ± 0.05	0.96 ± 0.05	0.09 ± 0.01	0.12 ± 0.01	1.27 ± 0.27	1.28 ± 0.03	77.2 ± 7.2	59.0 ± 0.6
5	Bussard	1.69 ± 0.07	1.69 ± 0.03	6.28 ± 0.28	6.85 ± 0.18	0.97 ± 0.05	1.07 ± 0.03	2.43 ± 0.12	2.27 ± 0.02	71.9 ± 1.8	56.3 ± 0.5
6	Constance	0.46 ± 0.03	0.60 ± 0.02	0.93 ± 0.05	1.54 ± 0.06	0.14 ± 0.01	0.20 ± 0.01	1.37 ± 0.15	1.49 ± 0.01	81.1 ± 3.5	57.9 ± 0.1
7	Dinosor	0.78 ± 0.01	0.96 ± 0.04	1.93 ± 0.01	2.96 ± 0.19	0.25 ± 0.01	0.41 ± 0.03	1.42 ± 0.01	1.50 ± 0.06	70.0 ± 0.8	54.8 ± 0.2
8	Enorm	0.92 ± 0.02	1.21 ± 0.01	2.43 ± 0.09	3.76 ± 0.06	0.35 ± 0.01	0.57 ± 0.01	1.68 ± 0.03	1.90 ± 0.03	74.7 ± 1.2	64.3 ± 0.3
9	Ephoros	0.94 ± 0.01	1.16 ± 0.01	2.70 ± 0.04	3.97 ± 0.05	0.38 ± 0.01	0.61 ± 0.01	1.78 ± 0.01	2.00 ± 0.01	73.3 ± 0.6	61.4 ± 0.5
10	Kodex	0.90 ± 0.01	1.14 ± 0.03	2.33 ± 0.05	3.54 ± 0.12	0.33 ± 0.01	0.53 ± 0.02	1.75 ± 0.01	1.85 ± 0.01	77.9 ± 0.8	62.4 ± 0.2
11	Melkior	0.57 ± 0.03	0.71 ± 0.01	1.22 ± 0.07	1.84 ± 0.01	0.16 ± 0.01	0.24 ± 0.01	1.38 ± 0.03	1.32 ± 0.01	72.0 ± 0.1	55.6 ± 0.1
12	Quebon	0.97 ± 0.03	1.18 ± 0.03	2.59 ± 0.09	3.86 ± 0.12	0.36 ± 0.02	0.59 ± 0.02	1.73 ± 0.01	1.94 ± 0.04	71.0 ± 0.4	62.6 ± 0.8
13	Rosario	1.27 ± 0.02	1.47 ± 0.03	3.78 ± 0.05	5.27 ± 0.12	0.54 ± 0.01	0.81 ± 0.02	1.82 ± 0.03	2.01 ± 0.02	68.4 ± 0.7	57.1 ± 0.4
14	Rustic	0.73 ± 0.01	0.88 ± 0.00	1.60 ± 0.03	2.34 ± 0.01	0.22 ± 0.01	0.32 ± 0.01	1.42 ± 0.01	1.43 ± 0.02	81.5 ± 1.3	63.0 ± 0.5
15	Samurai	0.69 ± 0.05	0.95 ± 0.02	1.60 ± 0.16	3.03 ± 0.07	0.23 ± 0.01	0.44 ± 0.01	1.56 ± 0.08	1.87 ± 0.03	71.6 ± 4.6	56.5 ± 0.7
16	Sokrates	0.80 ± 0.01	1.22 ± 0.01	1.85 ± 0.03	3.56 ± 0.05	0.24 ± 0.01	0.48 ± 0.01	1.26 ± 0.01	1.38 ± 0.03	66.7 ± 0.3	52.5 ± 0.6
17	Tuareg	0.94 ± 0.01	1.04 ± 0.04	2.15 ± 0.08	2.91 ± 0.11	0.29 ± 0.01	0.41 ± 0.02	1.43 ± 0.01	1.46 ± 0.06	72.3 ± 1.8	54.2 ± 1.6
	Min.	*1.69*	*1.69*	*0.64*	*1.54*	*0.09*	*0.12*	*1.26*	*1.28*	*66.7*	*52.5*
	Max.	*0.38*	*0.44*	*6.28*	*6.86*	*0.97*	*1.07*	*2.43*	*2.27*	*81.5*	*65.2*

[*] J_0: instantaneous compliance, J_1, J_2: retarded elastic compliances, r_1, r_2: retardation times

[**] Mean and standard deviation based on three repetitions

Annex 6. Influence of mixer type and mixing time (MT) on bread quality*

MT (min)	Pan bread					Plate bread		
	Dough Height (mm)	Bread Height (mm)	Oven rise (%)	Volume (cm^3)	Weight (g)	Volume (cm^3)	Weight (g)	H/W (-)
Pin mixer								
1.5	65.9 ± 1.4^{a}	72.7 ± 0.3^{a}	10.4 ± 2.6^{a}	349 ± 4^{a}	102.3 ± 0.2^{a}	311 ± 15^{a}	82.0 ± 0.3^{a}	0.67 ± 0.03^{a}
2	$67.7 \pm 1.3^{a,b}$	77.2 ± 0.7^{a}	14.1 ± 1.8^{a}	392 ± 3^{b}	$102.1 \pm 0.6^{a,b}$	353 ± 8^{b}	$81.4 \pm 0.5^{a,b}$	0.69 ± 0.01^{a}
3	$73.0 \pm 2.6^{b,c}$	82.8 ± 3.3^{b}	13.4 ± 0.6^{a}	$423 \pm 21^{b,c}$	$101.2 \pm 0.5^{a,b}$	$377 \pm 8^{b,c}$	80.5 ± 0.3^{b}	0.68 ± 0.01^{a}
4	$72.3 \pm 2.0^{a,b,c}$	84.1 ± 0.9^{b}	16.4 ± 2.2^{a}	$436 \pm 12^{c,d}$	101.0 ± 0.1^{b}	$408 \pm 7^{c,e}$	80.5 ± 0.3^{b}	0.70 ± 0.01^{a}
6	75.8 ± 0.5^{c}	87.2 ± 1.6^{b}	15.1 ± 2.7^{a}	459 ± 14^{d}	99.9 ± 0.5^{b}	443 ± 9^{d}	80.0 ± 0.4^{b}	0.73 ± 0.04^{a}
8	$73.7 \pm 5.0^{b,c}$	85.9 ± 2.0^{b}	16.7 ± 5.4^{a}	$443 \pm 9^{c,d}$	$97.9 \pm 3.4^{a,b}$	$427 \pm 23^{d,e}$	$78.5 \pm 2.2^{a,b}$	0.73 ± 0.04^{a}
Z-blade mixer								
2	68.5 ± 1.9^{a}	74.6 ± 1.1^{a}	8.9 ± 1.7^{a}	363 ± 9^{a}	102 ± 0.2^{a}	326 ± 10^{a}	81.0 ± 0.3^{a}	0.65 ± 0.02^{a}
4	73.9 ± 0.9^{b}	83.3 ± 1.0^{b}	$12.8 \pm 2.0^{a,b}$	426 ± 10^{b}	$101.2 \pm 0.6^{a,b}$	383 ± 29^{b}	$80.5 \pm 0.4^{a,b}$	$0.67 \pm 0.01^{a,b}$
6	74.9 ± 1.7^{b}	$85.5 \pm 1.3^{b,c}$	$14.2 \pm 3.1^{a,b}$	448 ± 3^{b}	$100.3 \pm 0.7^{b,c}$	$403 \pm 6^{b,c}$	$80.1 \pm 0.4^{a,b}$	$0.68 \pm 0.01^{a,b}$
10	76.7 ± 1.2^{b}	88.2 ± 0.9^{d}	15.0 ± 1.9^{b}	453 ± 16^{b}	$99.7 \pm 0.6^{b,c}$	428 ± 15^{c}	$80.1 \pm 0.4^{a,b}$	0.72 ± 0.03^{b}
14	75.4 ± 1.0^{b}	$86 \pm 0.4^{c,d}$	$14.2 \pm 2.1^{a,b}$	441 ± 9^{b}	99.8 ± 0.3^{c}	$421 \pm 12^{b,c}$	79.8 ± 0.4^{b}	$0.68 \pm 0.03^{a,b}$

*Values indicated with different letters in the same column for one mixer type are significantly different (α=0.05)

Curriculum vitae

Filip Van Bockstaele werd geboren te Aalst op 21 juli 1981 en behaalde in 1999 het diploma Hoger Secundair Onderwijs, richting Wetenschappen-Wiskunde aan het Sint-Maarteninstituut te Aalst. In juli 2004 behaalde hij met onderscheiding het diploma van Bio-ingenieur scheikunde aan de Universiteit Gent. In juli 2008 behaalde hij tevens het diploma Geaggregeerde voor het secundair onderwijs (AILO toegepaste biologische wetenschappen).

Sinds oktober 2004 is hij verbonden aan de Vakgroep Levensmiddelenwetenschappen en -technologie van Hogeschool Gent als onderzoeksassistent gefinancierd door het Onderzoeksfonds van Hogeschool Gent. Naast het doctoraatsonderzoek dat werd uitgevoerd in samenwerking met het Laboratorium voor Levensmiddelentechnologie en -proceskunde, participeerde hij eveneens in de dienstverlening- en onderwijsactiviteiten van de vakgroep. Zo verzorgde hij ondermeer de praktische oefeningen voor de vakken levensmiddelen-chemie, instrumentele analyse en graantechnologie, en begeleidde hij verscheidene binnen- en buitenlandse thesisstudenten.

Zijn onderzoek heeft geleid tot meerdere publicaties in peer-reviewed wetenschappelijke tijdschriften en hij verzorgde meerdere voordrachten en posters op nationale en internationale congressen. Zijn paper voorgesteld op het AACC congres in 2008 met als titel '*Rheological properties of wheat flour dough and their relation with bread volume: creep-recovery and dynamic oscillation measurements*' werd beloond met de *Isydore Hlynka Best Student Paper Award*.

In 2010 organiseerde hij in samenwerking met de AACC Rheology Division en het Laboratorium voor Levensmiddelentechnologie en –proceskunde, een succesvolle workshop te Gent met als topic '*Structure and rheology of cereal-based foods*'.

Relevant publications in international peer-reviewed journals

Van Bockstaele, F., De Leyn, I, Eeckhout, M. and Dewettinck, K. (2008). Rheological properties of wheat flour dough and the relationship with bread volume. I. Creep-recovery measurements. Cereal Chemistry, 85(6), 753-761.

Van Bockstaele, F., De Leyn, I, Eeckhout, M. and Dewettinck, K. (2008). Rheological properties of wheat flour dough and the relationship with bread volume. II. Dynamic oscillation measurements. Cereal Chemistry, 85(6), 762-768.

Van Bockstaele, F., De Leyn, I, Eeckhout, M. and Dewettinck, K. Non-linear creep-recovery measurements as a tool for evaluating the viscoelastic properties of wheat flour dough. Journal of Food Engineering, Submitted.

Van Bockstaele, F., De Leyn, I, Eeckhout, M., Meesen, G., Van Oostveldt, P. & Dewettinck, K. Influence of the mixing process on dough rheology and microstructure, Journal of Cereal Science, Submitted.

Other publications in international peer reviewed journals

Dewettinck, K., Van Bockstaele, F., Kuhne, B., de Walle, D. V., Courtens, T. M., and Gellynck, X. (2008). Nutritional value of bread: Influence of processing, food interaction and consumer perception. Journal of Cereal Science 48, 243-257.

Gellynck, X., Kuhne, B., Van Bockstaele, F., Van de Walle, D. and Dewettinck, K. (2009). Consumer perception of bread quality. Appetite, 53, 16-23.

Presentations at national and international conferences

Van Bockstaele, F., De Leyn, I., Eeckhout, M. and Dewettinck, K. Flour quality and dough rheology. European Young Cereal Scientists and Technologists Workshop, Cereals and Europe, Gaziantep, Turkey, 5-7 July 2006.

Van Bockstaele, F., De Leyn, I., Eeckhout, M. and Dewettinck, K. Rheological properties of wheat flour dough and their relation with bread volume: creep-recovery and dynamic oscillation measurements. AACC International Annual Meeting, Honolulu, Hawaii, USA, 21-24 September 2008.

Van Bockstaele, F., De Leyn, I., Eeckhout, M. and Dewettinck, K. How fundamental rheology can predict bread volume. 100th AOCS Annual Meeting & Expo, Orlando, Florida, USA, 3-6 May 2009.

Van Bockstaele, F., De Leyn, I., Eeckhout, M. and Dewettinck, K. Effect of mixer type, mixing time and dough formulation on the rheological properties of bread dough. Young Cereal Scientists and Technologists Workshop, Cereals and Europe, Viterbo, Italy, 3-5 August 2009.

Van Bockstaele, F., Eeckhout, M. and Dewettinck, K. Rheology and microstructure of bread dough. Short course 'Rheology and structure of cereal-based foods', Ghent, Belgium, 28-29 April 2010.

Poster presentations at national and international conferences

Gellynck, X., Kuhne, B., Van Bockstaele, F., Van de Walle, D. and Dewettinck, K., Consumer perception of bread quality. 12th Congress of the EAAE 'People, Food and Environments: Global Trends and European Strategies', Ghent, Belgium, 26-29 August 2008.

Van Bockstaele, F., De Leyn, I., Eeckhout, M. and Dewettinck, K., Rheological properties of wheat flour dough in relation to bread volume. Food2Know Re$earch Seminar, Ghent, Belgium, 24 February 2009.

Van Bockstaele, F., De Leyn, I., Eeckhout, M. and Dewettinck, K. Influence of mixing process on dough rheological properties. AACC International Annual Meeting, Baltimore, USA, 13-16 September 2009.

Van Bockstaele, F., Storme, T., Meesen, G., Van Oostveldt, P., Eeckhout, M. and Dewettinck, K. Changes in microstructure and rheology of bread dough during breadmaking. Exchange Seminar 'Feed, Food and Health', Ghent, Belgium, 28 September 2010.

Scientific award

Isydore Hlynka Best Student Paper Award 2009 (AACC Rheology Division)